**PUBLIC NOTICE**

**APPROVAL OF**
**PCDD/PCDF CONFIRMATORY TEST PROTOCOL**
**AND NOTIFICATION OF TEST BURN**

**HEXION SPECIALTY CHEMICALS**
**ORGANIC CHLORIDE INCINERATORS NCIN-1 AND NCIN-2**

**PERMIT NUMBER LAD 980 622 104 / ACTIVITY NUMBER AI 87883**
**ST. CHARLES PARISH, LOUISIANA**

The Louisiana Department of Environmental Quality (LDEQ is reviewing the PCDD/PCDF Confirmatory Test Protocol for the Organic Chloride Incinerators NCIN-1 and NCIN-2, for **Hexion Specialty Chemicals, 16122 River Road, Post Office Box 10, Norco, LA 70079 in St. Charles Parish**.

After the Test Protocol has been approved by LDEQ, Hexion Specialty Chemicals will conduct the test burn, currently scheduled to begin **March 30, 2009**, and to end on or about **April 13, 2009**, in accordance with the approved test protocol. Hexion Specialty Chemicals will be performing the PCDD/PCDF Confirmatory Test to confirm compliance of the Organic Chloride Incinerators NCIN-1 and NCIN-2 with the Hazardous Waste Combustion Maximum Achievable Control Technology (HWC MACT) PCDD/PCDF emission standards. As required, the Continuous Monitoring Systems Performance Evaluation Test (CMS PET) will be performed prior to the Confirmatory Test.

In the case of unanticipated delays, the time period will be dictated by the nature of the delay and will be discussed with LDEQ.

The protocol describes how the test burn will be conducted to meet the 40 Code of Federal Regulations (CFR) Subpart EEE regulatory requirements. The results accumulated from the test burn will be used to **confirm compliance of the Organic Chloride Incinerators NCIN-1 and NCIN-2 with the HWC MACT PCDD/PCDF emission standards.**

A copy of the approved test plan is available for review and copying (all documents copied will be subject to a $0.25 charge per copy page) at the LDEQ Public Records Center, Room 1-127, 602 North 5th Street, Baton Rouge, Louisiana. Viewing hours are from 8:00 a.m. to 4:30 p.m., Monday – Friday, (except holidays). **The available information can also be accessed electronically on the Electronic Document Management System (EDMS) on the DEQ public website at www.deq.louisiana.gov**.

Additional copies are available for review at the St Charles Parish Library, Norco Branch, 197 Good Hope Street, Norco, LA 70079 and at the St. Charles Parish Library, East Regional Branch,100 River Oaks Drive, Destrehan, LA 70047.

Individuals or public interest groups who would like to obtain additional information regarding this scheduled test burn should address MICHAEL GUIDRY, HEXION SPECIALTY CHEMICALS, 16122 River Road, Post Office Box 10, Norco, LA 70079, (504) 472-6585; or JILL MARTIN, LDEQ, Office of Environmental Services (OES), Waste Permits Division, Post Office Box 4313, Baton Rouge, Louisiana 70821-4313, (225) 219-3455.

Persons wishing to be included on the LDEQ permit public notice mailing list or for other public participation related questions should contact the Public Participation Group in writing at LDEQ, P.O. Box 4313, Baton Rouge, LA 70821-4313, by email at deqmaillistrequest@la.gov or contact the LDEQ Customer Service Center at (225) 219-LDEQ (219-5337).

**Permit public notices including electronic access to the approved test plan** can be viewed at the LDEQ permits public notice webpage at www.deq.louisiana.gov/apps/pubNotice/default.asp and general information related to the public participation in permitting activities can be viewed at www.deq.louisiana.gov/portal/tabid/2198/Default.aspx.

Alternatively, individuals may elect to receive the permit public notices via email by subscribing to the LDEQ permits public notice List Server at www.doa.louisiana.gov/oes/listservpage/ldeq_pn_listserv.htm

**All correspondence should specify AI Number 87883, Permit Number LAD 980 622 104, and Activity Number PER20080005.**

**Scheduled Publication Date: Thursday, January 29, 2009.**

# ORGANIC CHLORIDE INCINERATORS NCIN-1 AND NCIN-2

## PCDD/PCDF CONFIRMATORY TEST PROTOCOL

**PREPARED FOR:**

HEXION™
Specialty Chemicals

16122 RIVER ROAD
P.O. BOX 10
NORCO, LOUISIANA 70079

**January 2009, Revision 1**
**November 2008, Revision 0**
**Focus Project No. 060804**

**PREPARED BY:**

*Focus Environmental, Inc.*
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January 15, 2009

Mr. Bijan Sharafkhani
Administrator, Waste Permits Division
Louisiana Department of Environmental Quality
P.O. Box 4313
Baton Rouge, Louisiana 70821-4313

Reference: Response to Notices of Deficiency
PCDD/PCDF Confirmatory Test Protocol
Liquid and Vapor Incinerators NCIN-1 and NCIN-2
Hexion Specialty Chemicals
16122 River Road
Norco, Louisiana 70079
EPA ID No. LAD 980 622 104
Agency Interest No. 87883

Dear Mr. Sharafkhani:

Enclosed please find three (3) hard copies of the revised PCDD/PCDF Confirmatory Test Protocol (Revision 1) for the liquid and vapor incinerators, NCIN-1 and NCIN-2, located at the Norco, Louisiana facility. The plan is revised based on Notice of Deficiency (NOD) comments dated December 19, 2008. Responses to the NOD comments are also enclosed.

The Hazardous Waste Combustor (HWC) Maximum Achievable Control Technology (MACT) rule at 40 CFR 63.1207(e)(1)(ii) requires periodic confirmatory testing of the PCDD/PCDF emissions. The original plan was submitted November 13, 2008, more than the minimum sixty (60) days before the test date as required by the rule. The rule notes that Administrator will notify Hexion of the approval or the intent to deny approval of the plan within 30 calendar days after receipt. Hexion would like to conduct this test in March 2009. Hexion welcomes comments at LDEQ's earliest convenience.

If you have any questions or comments, you may contact Michael J. Guidry at Hexion by phone at (504) 472-6585 or e-mail at michael.guidry@hexion.com, or me by phone at (865) 692-8662 or e-mail at cemcbride@focusenv.com.

Sincerely,

Chris E. McBride
Consultant
Focus Environmental, Inc.

cc:

(1-hard copy and 1 electronic copy)
U.S. Environmental Protection Agency Region 6
Attn: Kishor Fruitwala
1445 Ross Avenue, Suite 1200
Mail Code:6PDA
Dallas, TX 75202-2733

Item No.1 – General Regulation
Comment: A Quality Assurance Project Plan (QAPP) was not submitted with the test protocol. A QAPP must be submitted or a previously approved applicable QAPP must be referenced in the written responses to the NODs. If a previously approved QAPP is referenced, an updated QAPP signature page, an updated organizational chart, and any other necessary updates must be submitted

Response: In the original version of the PCDD/PCDF Confirmatory Test Plan submitted in November 2008, the key QA/QC elements associated with the PCDD/PCDF sampling and analyses were included as Sections 4.0 and 5.0. In response to this comment, these sections of the PCDD/PCDF Confirmatory Test Plan have been removed and a separate QAPP prepared. Hexion has selected TestAmerica, Knoxville, Tennessee laboratory to prepare the sampling resins and perform the Method 0023A sample analyses. TestAmerica is LELAP accredited. Hexion has not yet selected the stack sampling contractor, however only LELAP accredited companies will be considered. When the stack sampling contractor is selected, the signed QAPP signature page can be provided.

Item No. 2 – General Regulation
Comment: The samplers chosen to perform the stack sampling procedures must be Louisiana Environmental Laboratory Accreditation Program (LELAP) accredited in all stack gas methods performed. The written responses to the NODs must state that the samplers chosen to perform the stack sampling procedures will be LELAP accredited in all stack gas methods performed.

Response: Please refer to the response to Comment 1.

Item No. # - 40 CFR 63.1207(e)(l)(ii)
Comment: A Continuous Monitoring System (CMS) Performance Evaluation Test Plan was not submitted with the test protocol as required by 40 CFR 63.1207(e)(l)(ii). A CMS Performance Evaluation Test Plan must be submitted to resolve this issue.

Response: A CMS PETP was provided with the Comprehensive Performance Test (CPT) plan submitted to LDEQ in October 2008. A copy of this same plan is added as Appendix B of the Confirmatory Test Plan. Hexion plans to conduct the CPT during calendar year 2009. Hexion plans to use the PCDD/PCDF emissions results from the PCDD/PCDF Confirmatory Test to be conducted in March 2009 as data-in-lieu to demonstrate compliance with the PCDD/PCDF emissions standard. Additionally, Hexion's desire is perform the CMS PETP one time, either during the PCDD/PCDF Confirmatory Test or the CPT, and use the CMS PET report to satisfy both test programs. Concurrence from LDEQ with this proposal is requested.

# ORGANIC CHLORIDE INCINERATORS
# NCIN-1 AND NCIN-2
# PCDD/PCDF CONFIRMATORY TEST PROTOCOL

**PREPARED FOR:**

HEXION™
Specialty Chemicals

**16122 RIVER ROAD**
**P.O. Box 10**
**NORCO, LOUISIANA 70079**

**January 2009, Revision 1**
**November 2008, Revision 0**
**Focus Project No. 060804**

**PREPARED BY:**

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# ORGANIC CHLORIDE INCINERATORS NCIN-1 AND NCIN-2 PCDD/PCDF CONFIRMATORY TEST PROTOCOL

## HEXION SPECIALTY CHEMICALS, INC.

## EPA I.D. NO. LAD 980 622 104

**SUBMITTED TO:**

HEXION™
Specialty Chemicals

16122 RIVER ROAD
P.O. BOX 10
NORCO, LOUISIANA 70079

JANUARY 2009, REVISION 1
NOVEMBER 2008, REVISION 0
FOCUS PROJECT NO. 060804

PREPARED BY:

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## List of Acronyms

| | |
|---|---|
| ACHE | allyl chloride heavy ends |
| ASTM | American Society for testing and materials |
| AWFCO | Automatic Waste Feed Cutoff |
| CAA | Clean Air Act |
| CATOX | catalytic oxidation |
| CEM | Continuous Emissions Monitoring |
| CFR | Code of Federal Regulations |
| CO | carbon monoxide |
| CPT | Comprehensive Performance Test |
| EPA | Environmental Protection Agency |
| FRS | MACT Final Replacement Standards |
| HCl | hydrogen chloride |
| HON | Hazardous Organic NESHAP |
| HRGC | high resolution gas chromatography |
| HRMS | high resolution mass spectrometry |
| HWC | Hazardous Waste Combustor |
| LELAP | Louisiana Environmental Laboratory Accreditation Program |
| MACT | Maximum Achievable Control Technology |
| MMBtu | million British thermal unit |
| NCIN-1 | Norco Organic Chloride Incinerator Number 1 |
| NCIN-2 | Norco Organic Chloride Incinerator Number 2 |
| NDIR | non-dispersive infrared |
| $O_2$ | oxygen |
| OPL | operating parameter limit |
| PCDD | polychlorinated dibenzo-p-dioxin |
| PCDF | polychlorinated dibenzofuran |
| QA | quality assurance |
| QC | quality control |
| RCRA | Resource Conservation & Recovery Act |
| RPP | Resolution Performance Products |
| SOP | standard operating procedure |
| TEQ | toxicity equivalents |
| USEPA | United States Environmental Protection Agency |

# 1.0 INTRODUCTION

## 1.1 FACILITY BACKGROUND INFORMATION

Hexion Specialty Chemicals, Inc. (Hexion) operates two hazardous waste incineration systems, Norco Organic Chloride Incinerators Numbers One and Two (NCIN-1 and NCIN-2), at the Norco, Louisiana chemical manufacturing facility. The units comply with the Hazardous Waste Combustor (HWC) Maximum Achievable Control Technology (MACT) rule. The HWC MACT rule at 40 CFR 63.1207 (e)(1)(ii) requires periodic confirmatory testing of the polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/PCDFs) emissions.

This plan presents the test protocol for confirming the continued compliance of NCIN-1 and NCIN-2 with the PCDD/PCDF emissions standard of less than 0.40 nanograms per dry standard cubic meter (ηg/dscm) 2,3,7,8-tetrachlorodibenzo-p-dioxin toxicity equivalents (TEQ) corrected to 7% oxygen [40 CFR 63.1203(a)(1)(i)(B) and 63.1219(a)(1)(i)(B)]. Hexion plans to conduct the required testing in March 2009.

## 1.2 UNIT OPERATIONS

NCIN-1 and NCIN-2 treat liquid hazardous wastes and vapor vent streams generated by the Hexion production operations. These streams include chlorinated organic compounds that form hydrogen chloride (HCl) when burned. The HCl is recovered from the combustion gas as aqueous HCl. The recovered HCl is used for neutralization in other Hexion operations or similar use by others. The incineration systems' combustion and energy recovery zones are identical. The incineration systems' downstream HCl recovery systems and air pollution control (APC) systems differ.

## 1.3 REGULATORY COMPLIANCE STATUS

The units currently operate under the provisions of Resource Conservation and Recovery Act (RCRA) and Clean Air Act (CAA) permits issued by the Louisiana Department of Environmental Quality (LDEQ). The units currently comply with the HWC MACT rule promulgated September 30, 1999 as amended (Interim Standards) February 13, 2002. The HWC MACT Final Replacement Standards (FRS) were promulgated October 12, 2005. Hexion will be demonstrating compliance with the FRS via a comprehensive performance test (CPT). Hexion plans to conduct the FRS CPT during calendar year 2009. Hexion plans to use the results from this confirmatory test as data-in-lieu to demonstrate compliance with the FRS PCDD/PCDF emissions standard.

# 2.0 TEST PROTOCOL

## 2.1 GENERAL

The PCDD/PCDF confirmatory testing will be performed on both NCIN-1 and NCIN-2. The incinerators will be operated at normal to high-normal liquid waste feed conditions, within the operating limits (OPLs) established by the 2004 CPT and summarized in Table 2-1. Testing of each incinerator will consist of three replicate sampling runs.

## 2.2 WASTE FEED CHARACTERISTICS

Hexion will conduct all testing while treating the allyl chloride heavy ends (ACHE) liquid waste stream. The ACHE represents the majority of liquid wastes treated in NCIN-1 and NCIN-2, and has a high chloride content.

## 2.3 OPERATING CONDITIONS

The target waste feed rate for the confirmatory test is 75% or greater of the maximum waste feed rate limit. Steady-state operating conditions will be achieved when the liquid waste feed rate, firebox temperature, and APC system operation have stabilized at the test target operating conditions for one hour.

## 2.4 SAMPLING AND ANALYSIS

This test will include the following sampling and analyses:

- Stack gas for PCDD/PCDF using a SW-846 Method 0023A sampling train
- Stack gas total hydrocarbon (THC) concentration using a temporary CEM according to the protocols in 40 CFR 60 Appendix A, Method 25A.
- Stack gas carbon monoxide (CO) and oxygen ($O_2$) concentrations by installed continuous emissions monitors (CEMs) according to the protocols in 40 CFR 60, Appendix B, Performance Specification 4B.

The selected stack sampling and analytical contractors will be accredited by the Louisiana Environmental Laboratory Accreditation Program (LELAP).

**Table 2-1. Current Operating Parameter Limits for NCIN-1 and NCIN-2**

| Operational Parameter | Current Operating Limit | | AWFCO | Averaging Period |
|---|---|---|---|---|
| | NCIN-1 | NCIN-2 | | |
| **Group 1 Parameters** | | | | |
| Maximum liquid waste feed rate (lb/hr) | 8,343 | 7,229 | Yes | Hourly Rolling Average |
| Minimum combustion temperature (° F) | 1,718 | 1,718 | Yes | Hourly Rolling Average |
| Minimum caustic scrubber recycle flow (gpm) (minimum L/G) | 550 | 410 | Yes | Hourly Rolling Average |
| Minimum caustic scrubber recycle pH | 9.20 | 8.22 | Yes | Hourly Rolling Average |
| Maximum caustic scrubber recycle conductivity (µS/cm) | 20,664 | 19,908 | Yes | 12-Hour Rolling Average |
| Minimum CATOX inlet gas temperature (° F) | 330 | 330 | Yes | Hourly Rolling Average |
| Maximum stack gas flow (mscfm) | 17.19 | 14.56 | Yes | Hourly Rolling Average |
| **Group 2 Parameters** | | | | |
| Maximum combustion chamber pressure (inwc) | 0.0 | 0.0 | Yes | None; 1-second delay |
| Maximum stack gas CO conc. (ppmv, dry @ 7% $O_2$) | 100 | 100 | Yes | Hourly Rolling Average |
| **Group 3 Parameters** | | | | |
| Minimum waste atomization steam pressure (psig) | 25 | 25 | No | None; checked daily |
| Minimum caustic scrubber recycle pressure (psig) | 13 | 54 | Yes | Hourly Rolling Average |
| Maximum CATOX inlet gas temperature (° F) | 700 | 700 | Yes | Hourly Rolling Average |

AWFCO - automatic waste feed cutoff
N/A-Not applicable

# 3.0 SAMPLING ANALYTICAL AND MONITORING PROCEDURES

## 3.1 GENERAL

The objective of this test program is to confirm compliance of NCIN-1 and NCIN-2 with the HWC MACT PCDD/PCDF emissions standards. The test program samples will be collected using the methods summarized in Table 3-1. The total numbers of field samples expected to be generated during the NCIN-1 and NCIN-2 incinerator testing are also summarized Table 3-1.

## 3.2 REFERENCE METHODS

The test program sampling and analytical reference sources include:

- Appendix A to 40 CFR 60, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60 (EPA)
- Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates (SW-846)
- American Society for Testing and Materials (ASTM) Annual Book of ASTM Standards.

Reference sampling and analytical methods are identified throughout this test protocol.

## 3.3 SAMPLING PROCEDURES

### 3.3.1.1 Stack Gas Method 0023A (PCDD/PCDF)

The stack gas will be sampled for PCDD/PCDFs using a SW-846 Method 0023A sampling train. The Method 0023A sampling train will be analyzed for PCDD/PCDFs via Method 8290 [high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS)]. At 40 CFR 63.1208(b)(1)(iii), the HWC MACT rule requires that the Method 0023A sampling train be operated for a minimum of 180 minutes (3 hours) to sample a minimum of 2.5 dry standard cubic meters of stack gas during each sampling run so that any 2,3,7,8-chlorinated PCDD/PCDF congener that is non-detect may be counted as zero. The Method 0023A sampling train will be operated for the minimum specified time and to obtain the minimum specified volume.

### 3.3.1.2 Stack Gas Method 25A (Hydrocarbons)

The stack gas will be continuously monitored for total hydrocarbons (THC) to demonstrate compliance with the HWC MACT performance standard 10 ppmdv @ 7% $O_2$. THC monitoring will be performed using a temporary CEMS. The temporary THC CEMS will be calibrated and operated in accordance with the procedures in 40 CFR 60 Appendix A, Method 25A. THC concentration will be reported as propane, corrected to 7% oxygen, dry basis.

### 3.4 ANALYTICAL PROCEDURES

The Method 0023A sample analysis will include separate preparation and analysis of the front-half (filter element and probe rinses) and back-half (XAD-2 resin trap and condenser rinses) sampling train fractions. PCDD/PCDF analyses will be performed following the protocols of SW-846 Method 8290.

### 3.5 MONITORING PROCEDURES

Continuous monitoring of the process operating variables noted in Table 2-1 will be performed. The operating data will be included in the final test report.

## Table 3-1. Sample Collection Methods, Equipment, and Frequency

| Sample Name | Sampling Reference Method | Sample Container | Analysis | General Procedure/Frequency | NCIN-1 Test 1 | NCIN-2 Test | Field QC | Total Field Samples |
|---|---|---|---|---|---|---|---|---|
| Method 0023A Particulate Filter | SW846 Method 0023A | Petri Dish | PCDD/PCDFs | Collect integrated sample for PCDD/PCDFs during each test run. | 3 | 3 | 1-blank train | 7 |
| Method 0023A Front Half Acetone and Methylene | | 500 mL glass sample bottle | PCDD/PCDFs | Sample for a minimum 180 minutes to sample a minimum of 3.0 dry standard cubic meters of stack gas. | 3 | 3 | 1-blank train | 7 |
| Method 0023A Front Half Toluene Rinses | | 250 mL glass sample bottle | PCDD/PCDFs | | 3 | 3 | 1-blank train | 7 |
| Method 0023A XAD-2 Resin | | Resin Trap | PCDD/PCDFs | | 3 | 3 | 1-blank train | 7 |
| Method 0023A Back Half Acetone and Methylene | | 500 mL glass sample bottle | PCDD/PCDFs | | 3 | 3 | 1-blank train | 7 |
| Method 0023A Back Half Toluene Rinses | | 250 mL glass sample bottle | PCDD/PCDFs | | 3 | 3 | 1-blank train | 7 |
| Method 0023A Acetone Reagent Blank | SW846 Method 0023A | 250 mL glass sample bottle | PCDD/PCDFs | Reagent Blank | -- | -- | 1 | 1 |
| Method 0023A Methylene Chloride Reagent Blank | SW846 Method 0023A | 250 mL glass sample bottle | PCDD/PCDFs | Reagent Blank | -- | -- | 1 | 1 |
| Method 0023A Toluene Reagent Blank | SW846 Method 0023A | 250 mL glass sample bottle | PCDD/PCDFs | Reagent Blank | -- | -- | 1 | 1 |
| PCDD/PCDF Audit Sample | SW846 Method 8290 | Ampoule with spiked XAD-2 Resin | PCDD/PCDFs | As provided by Agency | -- | -- | 1 | 1 |
| Carbon Dioxide and Oxygen by Orsat | EPA Methods 3 and 3A | 25-50 L Tedlar gas sample bag | Carbon Dioxide and Oxygen by Orsat | Collect two 1-hour integrated bag sample for on-site Orsat analysis. | 6 | 6 | -- | 12 |
| Total Hydrocarbons | EPA Methods 25A | Temporary Continuous Emissions Monitor | Total Hydrocarbons | Continuous during each test run | -- | -- | -- | -- |
| Carbon Monoxide | EPA Method 10; Performance Specification 4B | Installed Continuous Emissions Monitor | Carbon Monoxide | Continuous during each test run | -- | -- | -- | -- |
| Oxygen | EPA Method 3A; Performance Specification 4B | Installed Continuous Emissions Monitor | Oxygen | Continuous during each test run | -- | -- | -- | -- |
| **TOTAL SAMPLES** | | | | | | | | **58** |

# 4.0 TEST SCHEDULE

## 4.1 PLANNED TEST DATE

Hexion plans to conduct the confirmatory testing in March 2009. Hexion will notify LDEQ and EPA Region 6 at least 60 days before the planned date for starting the test. The test start date will be confirmed one week before the start of the test.

## 4.2 TEST DURATION AND DETAILED SCHEDULE TEST ACTIVITIES

The PCDD/PCDF confirmatory test will consist of three replicate sampling runs performed each of the incinerators . The PCDD/PCDF confirmatory sampling may begin once the incinerator being tested has reached the target test conditions. The order of testing will be determined at the time of the test. The daily schedule of testing is as follows:

- Monday-Test team travels to test site, participates in on-site health and safety training, and begins test equipment setup on the first unit to be tested.
- Tuesday-Complete test equipment set up on the first unit to be tested; conduct high-normal waste feed rate test on the first unit to be tested, Runs 1 and 2.
- Wednesday-Conduct high-normal waste feed rate test on the first unit to be tested, Run 3; begin relocation of test equipment to second unit to be tested.
- Thursday- Complete test equipment relocation and set up on the second unit to be tested; conduct high-normal waste feed rate test on the second unit to be tested, Runs 1 and 2.
- Friday- Conduct high-normal waste feed rate test on the second unit to be tested, Run 3; recover testing equipment and demobilize from test site.
- Saturday-Contingency day, if needed.

## 4.3 QUANTITY OF WASTE TO BE BURNED

The estimated hours of operation to complete testing are summarized in Table 4-1. The amount of liquid waste feed is summarized in Table 4-1.

## 4.4 TEST INTERRUPTIONS

In the event of an AWFCO or similar test interruption, all stack gas sampling will be suspended immediately. Stack sampling pumps will be switched off, but probes may remain in the stack.

Should the situation be resolved very shortly (15 minutes or less), and the waste feed instantaneous rate is resumed at or above 90% of the rate prior to the test stoppage event, and other target conditions are comparable to before the test interruption, stack sampling may be resumed at the discretion of the project manager and after consultation with the incinerator operations staff. Optionally, the project manager may

elect to hold off the re-start of sampling until the hourly rolling averages have re-established at or closer to test target values.

Should the situation take longer to be resolved (more than 15 minutes), all sampling will be suspended until the hourly rolling averages have re-established at or close to test target values. The stack gas sampling probes will be removed from the stack ports and the nozzles sealed off with Teflon tape. Stack sampling equipment will be maintained on hot standby pending a test re-start decision. Once the situation is corrected, waste feed has resumed, and the hourly rolling averages are re-established, testing will resume at the direction of the project manager.

Should the situation become evident that testing can not be resumed in 1-2 hours, or will take even longer to resolve, the project manager may suspend testing for the day. The project manager will assess whether any stack gas sampling trains that have been completed should be retained or discarded. Most incomplete sampling trains may be held for up to 24 hours and then resumed. If testing can be resumed the following day, the incomplete stack gas sampling trains and completed sampling train samples will be secured for the night in such manner as to properly preserve the samples. Sampling will resume the following day where testing the previous day ended once the unit is back at the target test conditions. Optionally, the test run may be scrubbed altogether with all samples to that point being discarded, and all stack gas sampling started anew when testing can be resumed. No incomplete sampling train samples will be held over for more than 24 hours.

All test start/stop/suspension/scrub decisions will be communicated to the regulatory observers present at the time of testing. Such decisions may include consultations between the project manager, the incinerator operations staff, and the regulatory observers present.

**Table 4-1. Quantity of Feed Materials for Testing**

| Parameter | Test Targets | Target Units | Totals | | | | | Total Units |
|---|---|---|---|---|---|---|---|---|
| | | | Run 1 | Run 2 | Run 3 | Contin-gency | Test | |
| **NCIN-1 Confirmatory Test** | | | | | | | | |
| Duration | Estimate | hours | 5 | 5 | 5 | 5 | 20 | hours |
| Waste Feed Rate | 6,257 | lb/hr | 31,286 | 31,286 | 31,286 | 31,286 | 125,145 | lbs |
| **NCIN-2 Confirmatory Test** | | | | | | | | |
| Duration | Estimate | hours | 5 | 5 | 5 | 5 | 20 | hours |
| Waste Feed Rate | 5,422 | lb/hr | 27,109 | 27,109 | 27,109 | 27,109 | 108,435 | lbs |

# QUALITY ASSURANCE PROJECT PLAN
# FOR THE
# ORGANIC CHLORIDE INCINERATORS NCIN-1 AND NCIN-2
# PCDD/PCDF CONFIRMATORY TEST
# HEXION SPECIALTY CHEMICALS, INC.
# EPA I.D. NO. LAD 980 622 104

**PREPARED FOR:**

HEXION™
Specialty Chemicals

16122 RIVER ROAD
P.O. BOX 10
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JANUARY 2009, Revision 0
FOCUS PROJECT NO. 060804

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## 1.0 QUALITY ASSURANCE PROJECT PLAN APPROVAL FORM AND DISTRIBUTION LIST

**Project Title:** Hexion Specialty Chemicals, Inc. (Hexion)
Organic Chloride Incinerators NCIN-1 AND NCIN-2
PCDD/PCDF Confirmatory Test
Norco, Louisiana

**Expected Comprehensive Performance Test (Confirmatory Test) Date:** March 2009

**Project Approvals:**

| Name/Function/Organization | Signature | Date |
|---|---|---|
| Michael J. Guidry<br>Hexion Test Project Manager<br>Hexion Specialty Chemicals, Inc. | | |
| Stack Sampling Coordinator | | |
| Dr. William C. Anderson<br>Laboratory Analysis Coordinator<br>Test America, Inc. | | |

QAPP Approval Statement: The individuals listed above:

1) Have received, read, and agreed to the appropriate information pertaining to assigned project responsibilities listed and provided in this QAPP, and

2) Agree that no testing methods will be modified. If modifications do occur to any sampling and analytical methods as specified in the associated PCDD/PCDF Confirmatory Test Plan, they are to be identified and explained in the sampling and analytical narratives, and discussed in the body of the test report.

QAPP Distribution List

| Project Organization/Title | Organization/Name | No. of Copies |
|---|---|---|
| Hexion Test Project Manager | Hexion Specialty Chemicals, Inc. | 1 |
| Stack Sampling Coordinator | | 1 |
| Laboratory Analysis Coordinator | Test America, Inc. | 1 |
| Louisiana Department of Environmental Quality | LDEQ | 3 |

The QAPP was distributed to these individuals, was read by the individuals, and they agree to the appropriate information pertaining to their project responsibilities as listed and provided in this QAPP.

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## ATTACHMENTS

## ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| CAR | corrective action request |
| CCB | continuing calibration blank |
| CCC | calibration check compound |
| CCV | continuing calibration verification |
| CEM | continuous emissions monitor |
| CEMS | continuous emissions monitoring system |
| CF | calibration factor |
| cfm | cubic feet per minute |
| CFR | Code of Federal Regulations |
| CLP | Contract Laboratory Program |
| CO | carbon monoxide |
| Confirmatory Test | comprehensive performance test |
| CMS | continuous monitoring system |
| COC | chain of custody |
| DI | deionized (water) |
| DNAPL | dense non-aqueous phase liquid |
| DOT | U.S. Department of Transportation |
| DQO | data quality objective |
| dscf | dry standard cubic foot |
| dscfm | dry standard cubic feet per minute |
| dscm | dry standard cubic meter |
| dscmm | dry standard cubic meters per minute |
| EDL | estimated detection limit |
| EPA | U.S. Environmental Protection Agency |
| FR | feed rate |
| HC | hydrocarbons |
| HRGC/HRMS | high resolution gas chromatography/ high resolution mass spectrometry |
| HWC | Hazardous Waste Combustor |
| IATA | International Air Transport Association |
| ICB | initial calibration blank |
| ICV | initial calibration verification |
| IDL | instrument detection limit |
| inwc | inches water column |
| kg | kilograms |
| L | liter |
| L/G | liquid to gas ratio |
| L/min | liters per minute |
| LAC | Laboratory Analysis Coordinator |
| lb or lbs | pounds |
| LCS | laboratory control standard |
| LCSD | laboratory control standard duplicate |
| LDEQ | Louisiana Department of Environmental Quality |
| LELAP | Louisiana Environmental Laboratory Accreditation Program |

| | |
|---|---|
| $m^3$/min | cubic meters per minute |
| MACT | Maximum Achievable Control Technology |
| MDL | method detection limit |
| mg | milligrams |
| mg/L or mg/l | milligrams/liter |
| µg or ug | micrograms |
| µg/$m^3$ or ug/$m^3$ | micrograms/cubic meter |
| min | minute |
| mL or ml | milliliters |
| mm | millimeters |
| NA | not applicable |
| ND | not detect |
| ηg or ng | nanograms |
| $O_2$ | oxygen |
| OSW | EPA Office of Solid Waste |
| PCDD | polychlorinated dibenzo-p-dioxin |
| PCDF | polychlorinated dibenzofuran |
| PE | performance evaluation |
| pg | picograms |
| ppm | parts per million |
| ppmv or ppmdv | parts per million dry volume |
| QA | quality assurance |
| QAPP | Quality Assurance Project Plan |
| QC | quality control |
| RF | response factor |
| RFA | request for analysis |
| RPD | relative percent difference |
| RRF | relative response factor |
| RRT | relative retention time |
| RSD | relative standard deviation |
| RT | retention time |
| SOP | standard operating procedure |
| SW846 | "Test Methods for Evaluating Solid Wastes Physical/Chemical Methods (SW-846)," Third Edition, 1986 and updates (December 1997). |
| 2,3,7,8-TCDD | 2,3,7,8-tetrachlorodibenzo-p-dioxin |
| TEQ | toxicity equivalents |
| TEF | toxicity equivalents factors |
| wc | water column |
| WM | wide-mouth |

# 3.0 PROJECT DESCRIPTION

## 3.1 GENERAL

Hexion Specialty Chemicals, Inc. (Hexion) operates two hazardous waste incineration systems, Norco Organic Chloride Incinerators Numbers One and Two (NCIN-1 and NCIN-2), at the Norco, Louisiana chemical manufacturing facility. The units comply with the Hazardous Waste Combustor (HWC) Maximum Achievable Control Technology (MACT) rule. The HWC MACT rule at 40 CFR 63.1207 (e)(1)(ii) requires periodic confirmatory testing of the polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/PCDFs) emissions. This Quality Assurance Project Plan (QAPP) is an integral part of the PCDD/PDCF Confirmatory Test Plan for NCIN-1 and NCIN-2, and must be used in conjunction with this plan. This section provides a brief description of the test objectives, the unit operating protocol, the associated sampling and analytical protocol, and how they relate to operation of NCIN-1 and NCIN-2. Refer to the Confirmatory Test Plan for more detailed information on the scope of the testing.

The applicable PCDD/PCDF emissions standard is less than 0.40 nanograms per dry standard cubic meter (ηg/dscm) 2,3,7,8-tetrachlorodibenzo-p-dioxin toxicity equivalents (TEQ) corrected to 7% oxygen [40 CFR 63.1203(a)(1)(i)(B) and 63.1219(a)(1)(i)(B)]. The Confirmatory Test will confirm that NCIN-1 and NCIN-2 continue to operate in compliance with this emissions standard. The selected stack sampling and analytical contractors will be accredited by the Louisiana Environmental Laboratory Accreditation Program (LELAP).

## 3.2 QUALITY ASSURANCE PROJECT PLAN SCOPE

This QAPP presents the organization, objectives, functional activities, and specific Quality Assurance (QA) and Quality Control (QC) activities for the Confirmatory Test of NCIN-1 and NCIN-2. This QAPP also describes the specific QA/QC protocols that will be followed for sampling, sample handling and storage, chain-of-custody, and laboratory analysis during the test program.

All QA/QC procedures will be in accordance with applicable professional technical standards, government regulations and guidelines, and specific project goals and requirements. The test plans and this associated QAPP have been prepared in accordance with EPA hazardous waste thermal treatment system testing and QAPP guidance documents, in particular the following:

- EPA Requirements for Quality Assurance Project Plans (EPA QA/R-5 EPA/240/B-01/003), March 2001

- Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (QAMS-005/80)
- Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration, EPA/625/6-89/023, January 1990.
- National Emission Standards for Hazardous Air Pollutants from Hazardous Waste Combustors, 40 CFR 63 Subpart EEE, October 12, 2005.
- Rules and Regulations of the State of Louisiana applicable to operators of hazardous waste incinerators [LAC 33:V.3111].
- EPA, "New Source Performance Standards, Test Methods and Procedures," Appendix A, 40 CFR 60.
- EPA, "Test Methods for Evaluating Solid Wastes Physical/Chemical Methods (SW-846)", Third Edition, 1986 and updates (December 1997).

## 3.3 UNIT OPERATIONS

NCIN-1 and NCIN-2 treat liquid hazardous wastes and vapor vent streams generated by the Hexion production operations. These streams include chlorinated organic compounds that form hydrogen chloride (HCl) when burned. The HCl is recovered from the combustion gas as aqueous HCl. The recovered HCl is used for neutralization in other Hexion operations or similar use by others. The incineration systems' combustion and energy recovery zones are identical. The incineration systems' downstream HCl recovery systems and air pollution control (APC) systems differ.

## 3.4 REGULATORY COMPLIANCE STATUS

The units currently operate under the provisions of Resource Conservation and Recovery Act (RCRA) and Clean Air Act (CAA) permits issued by the Louisiana Department of Environmental Quality (LDEQ). The units currently comply with the HWC MACT rule promulgated September 30, 1999 as amended (Interim Standards) February 13, 2002. The HWC MACT Final Replacement Standards (FRS) were promulgated October 12, 2005. Hexion will be demonstrating compliance with the FRS via a comprehensive performance test (CPT). Hexion plans to conduct the FRS CPT during calendar year 2009. Hexion plans to use the results from this confirmatory test as data-in-lieu to demonstrate compliance with the FRS PCDD/PCDF emissions standard.

## 3.5 OPERATING CONDITIONS

The PCDD/PCDF confirmatory testing will be performed on both NCIN-1 and NCIN-2. The incinerators will be operated at normal to high-normal liquid waste feed conditions, within the operating limits (OPLs) established by the 2004 CPT and summarized in Table 2-1. The target waste feed rate for the confirmatory test is 75% or greater of the maximum waste feed rate limit. Steady-state operating conditions will be achieved when the liquid waste feed rate, firebox temperature, and APC system operation have stabilized at the test target operating conditions for one hour.

## 3.6 SAMPLING AND ANALYSIS

This test will include the following sampling and analyses:

- Stack gas for PCDD/PCDF using a SW-846 Method 0023A sampling train
- Stack gas total hydrocarbon (THC) concentration using a temporary CEM according to the protocols in 40 CFR 60 Appendix A, Method 25A.
- Stack gas carbon monoxide (CO) and oxygen ($O_2$) concentrations by installed continuous emissions monitors (CEMs) according to the protocols in 40 CFR 60, Appendix B, Performance Specification 4B.

Testing of each incinerator will consist of three replicate sampling runs.

**Table 3-1. Current Operating Parameter Limits for NCIN-1 and NCIN-2**

| Operational Parameter | Current Operating Limit | | AWFCO | Averaging Period |
|---|---|---|---|---|
| | NCIN-1 | NCIN-2 | | |
| **Group 1 Parameters** | | | | |
| Maximum liquid waste feed rate (lb/hr) | 8,343 | 7,229 | Yes | Hourly Rolling Average |
| Minimum combustion temperature (° F) | 1,718 | 1,718 | Yes | Hourly Rolling Average |
| Minimum caustic scrubber recycle flow (gpm) (minimum L/G) | 550 | 410 | Yes | Hourly Rolling Average |
| Minimum caustic scrubber recycle pH | 9.20 | 8.22 | Yes | Hourly Rolling Average |
| Maximum caustic scrubber recycle conductivity (µS/cm) | 20,664 | 19,908 | Yes | 12-Hour Rolling Average |
| Minimum CATOX inlet gas temperature (° F) | 330 | 330 | Yes | Hourly Rolling Average |
| Maximum stack gas flow (mscfm) | 17.19 | 14.56 | Yes | Hourly Rolling Average |
| **Group 2 Parameters** | | | | |
| Maximum combustion chamber pressure (inwc) | 0.0 | 0.0 | Yes | None; 1-second delay |
| Maximum stack gas CO conc. (ppmv, dry @ 7% $O_2$) | 100 | 100 | Yes | Hourly Rolling Average |
| **Group 3 Parameters** | | | | |
| Minimum waste atomization steam pressure (psig) | 25 | 25 | No | None; checked daily |
| Minimum caustic scrubber recycle pressure (psig) | 13 | 54 | Yes | Hourly Rolling Average |
| Maximum CATOX inlet gas temperature (° F) | 700 | 700 | Yes | Hourly Rolling Average |

# 4.0 ORGANIZATION OF PERSONNEL, RESPONSIBILITIES, AND QUALIFICATIONS

## 4.1 GENERAL

The project organization for this test is summarized in Figure 4-1. The Hexion Plant Manager is responsible for oversight of all activities performed at the Hexion site. The Hexion Environmental, Health & Safety Manager and the Hexion Test Project Manager report to the Hexion Plant Manager.

The Hexion Environmental, Health & Safety Manager is responsible for all day-to-day environmental compliance activities related to the plant site. During the testing, the Hexion Environmental, Health & Safety Manager and other environmental staff members will be available to lend support to the testing program where needed.

During the test, the Hexion Test Project Manager, on behalf of the Hexion Environmental, Health & Safety Manager, will be responsible for ensuring that the processes run properly and that the unit achieves the desired test conditions on each test day. As such, the Hexion Test Project Manager, working through the Hexion Production Manager, will assign responsibilities concerning unit operations. The Hexion Test Project Manager will be responsible for ensuring that all of the applicable process data are collected during each of the test runs. The Hexion Test Project Manager will also be responsible for supervising all of the testing contractors associated with the program, and will serve as the official communication link between Hexion and the respective contractors and regulatory observers.

The Stack Sampling Contractor is experienced in conducting the stack sampling called for in the test plan and will conduct the stack sampling for this project. The Contract Analytical Laboratory is experienced in the analysis of stack emissions samples, and will provide analytical services for this project. Both of these selected contractors will be accredited by the Louisiana Environmental Laboratory Accreditation Program (LELAP).

## 4.2 TEST PROJECT MANAGER

The Hexion Test Project Manager is responsible for the execution of the NCIN-1 and NCIN-2 Confirmatory Test Plan. During the test, the Hexion Test Project Manager is responsible for the overall implementation of the test program. The Hexion Test Project Manager will manage the sampling contractors during testing. Specific Hexion Test Project Manager responsibilities include:

- Ensuring compliance with the test plan by the stack samplers
- Documenting testing activities via a field log

- Assisting and interfacing with the regulatory observers and/or oversight contractors during the test
- Providing coordination between the stack sampling team during the test, and the NCIN-1 and NCIN-2 operations, especially regarding decisions to start, stop, hold or repeat sampling runs

### 4.3 STACK SAMPLING TEAM COORDINATOR

The Stack Sampling Coordinator have overall responsibility for the collection and handling of all stack gas related samples. The Stack Sampling Coordinator will implement the QAPP and prepare the final test report. The Stack Sampling Coordinator has the following responsibilities:

- Preparing and shipping stack sampling equipment, and shipping containers to the test site
- Preparing and calibrating stack sampling equipment
- Directing and/or participating in stack sampling activities
- Recording field test data required by the test plans and stack sampling methods
- Reviewing and approving stack sample collection sheets and stack sampling field data sheets
- Overseeing recovery of stack sampling-related samples and preservation of those samples
- Providing field review of process operating logs, and completed sample collection sheets, stack sampling logs, and Chain of Custody (COC) and Request for Analysis (RFA) forms
- Reducing stack sampling data and performing all calculations and QA activities required by the stack sampling methods
- Interfacing with the Laboratory Analysis Coordinator while samples are being analyzed
- Preparing a draft and final report of stack sampling activities.
- Certifying the overall test results and the final test report.

### 4.4 HEXION PROCESS OPERATIONS

The Hexion Process Operations will be responsible for the operation of the incinerators. Their duties will include:

- Maintaining the incinerators within specified target limits
- Maintaining logs of process data as required
- Downloading and providing the NCIN-1 and NCIN-2 one-minute operating data to the Hexion Test Project Manger in Microsoft Excel or ASCII format
- Assisting in the logistics of the stack sampling team where necessary.

### 4.5 LABORATORY ANALYSIS COORDINATOR

The Laboratory Analysis Coordinator will have overall responsibility for the analysis of the stack gas samples. The Laboratory Analysis Coordinator has the following responsibilities:

- Receiving, verifying, and documenting that incoming field samples correspond to the sample COC and RFA information

- Notifying the Hexion Test Project Manager and the Stack Sampling Coordinator of any discrepancies or problems in the COC and RFA information, preservation, or sample condition
- Maintaining records of incoming samples
- Tracking samples through processing, analysis, and disposal
- Designating QC samples for analysis during the project
- Verifying that laboratory personnel are trained and qualified in specified laboratory QC and analytical procedures
- Verifying that laboratory QC and analytical procedures are being followed as specified in this QAPP, the laboratory specific QA/QC Plan, and the laboratory specific analytical standard operating procedures (SOPs)
- Reviewing QC and sample data during analysis and determining if repeat analyses are needed
- Submitting certified QC and sample analysis results and data packages to the Hexion Test Project Manager
- Notifying the Hexion Test Project Manager and the Stack Sampling Coordinator of any QC excursions during the preparation and analysis of the field samples or associated QC samples
- Reviewing all analytical data and preparing a statement in the certificate of analysis summary regarding if any of the test data are invalid or unusable
- Approving via affixing signature to all complete analytical data packages.
- Archiving analytical data.

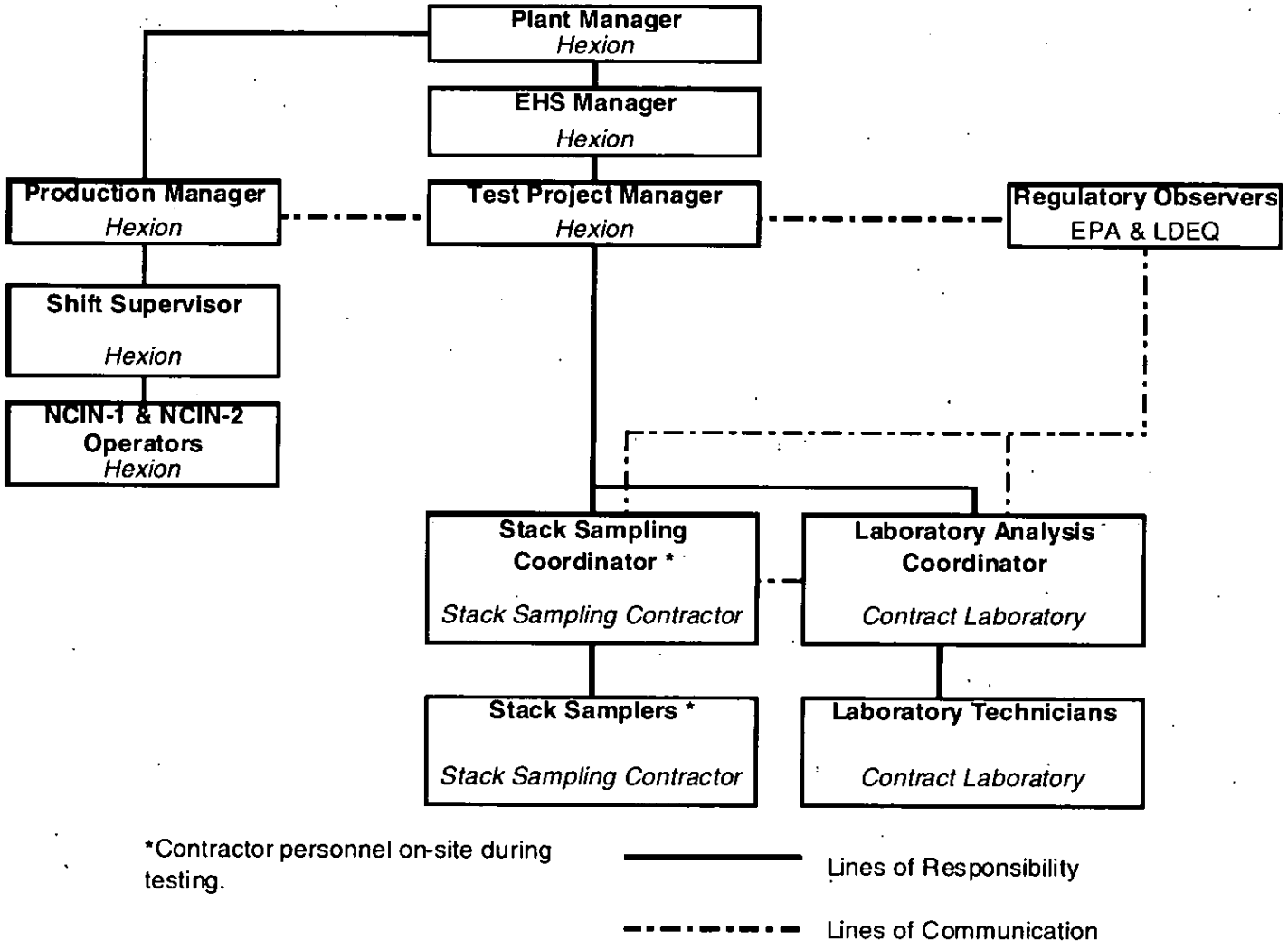


**Figure 4-1. Hexion PCDD/PCDF Confirmatory Test Project Organization and Responsibility**

# 5.0 QUALITY ASSURANCE OBJECTIVES AND QUALITY CONTROL OBJECTIVES

## 5.1 GENERAL

The overall quality assurance objective is to produce a complete PCDD/PCDF data set that can be used to fully assess and validate the operation of NCIN-1 and NCIN-2 Furnace relative to the HWC MACT PCDD/PCDF emissions standard. The selected stack sampling contractor and the contract analytical laboratory are LELAP certified and well experienced in the types of sampling and analyses represented by this test program. The analytical laboratory will provide the best and lowest PCDD/PCDF detection limits possible. Agency issued audit samples, as may be provided, will be included in the analysis scope as a third party check of the laboratory accuracy.

Table 5-1 presents target data quality objectives (DQOs) for precision and accuracy for the PCDD/PCDF analyses that will be performed during the test program. QA/QC objectives for precision, accuracy, representativeness, completeness, and comparability are defined in this section. Procedures and formulas for determining accuracy and precision are presented in Section 13.0 of this document. The following definitions briefly describe the meaning of each QA/QC objective:

**Precision:** A measure of mutual agreement among individual measurements of the same property, usually under "prescribed similar conditions". Various measures of precision exist depending on the prescribed similar conditions. If the number of samples is less than three, the precision is described as range percent or relative percent difference (RPD) from the average of replicate measured values for analysis of the same parameter. If the number of samples is three or greater, precision is best described in terms of relative standard deviation (RSD).

**Accuracy:** The degree of agreement of a measurement (or an average of measurements of the same parameter) X, with an accepted reference or true value, T. Accuracy is usually expressed as the difference between the two values, X - T, or the difference as a percentage of the reference or true value, 100 (X - T)/T, and sometimes expressed as a ratio, X/T. In some cases, accuracy is described as the percentage recovery of a known quantity of material added to a sample prior to analysis. Accuracy is a measure of the bias in a system.

**Completeness:** A measure of the amount of valid data obtained compared to the amount expected to be collected under normal conditions. Completeness is usually expressed as a percentage.

**Representativeness:** The degree to which data accurately and precisely represent a characteristic of a population, parameter variation at a sampling point, process condition, or an environmental condition.

**Comparability:** The confidence with which one set of data can be compared to another.

## 5.2 PRECISION AND ACCURACY

All sampling and analytical activities will be conducted following referenced procedures. All reference materials used as calibration standards, surrogate compounds, or laboratory control samples will be of the highest purity commercially available. The calibration of instruments used during analysis will be verified each day that samples are analyzed as described in later sections of this QAPP. Assessment of data precision and accuracy will be accomplished by evaluating the results from multiple analyses of the same parameter, and surrogate spiked samples. Field and laboratory contamination will be assessed through the analysis of reagent, instrument, method, and field and trip blanks.

Precision estimates presented in Table 5-1 represent variability for replicate measurements of the same parameters, expressed in terms of relative percent difference (RPD) and relative standard deviation (RSD).

Accuracy values in Table 5-1 include components of both random error and bias, expressed as a percentage of the "true" of "known" value (for reference materials) or percent analyte recovery (for spiked samples). The QA/QC program will focus upon controlling measurement error within the estimated limits of measurement uncertainty as specified in Table 5-1. QA/QC determinations which fall outside of the target range will be flagged and an assessment of the impact, if any, on the usefulness of the data on the overall results and conclusions of the test program will be provided in the final test report.

The analytical laboratory's LELAP-approved standard operating procedures (SOPs) for each analysis will be followed. If ongoing QA/QC procedures reveal that a measurement's error has exceeded the estimated data quality limits, the source of the excessive error will be identified and corrective action will be taken, as described in Section 14.0. If data fall outside the acceptable range of precision and accuracy, even after corrective action has been taken, those data points will be flagged in the final report. The precision and accuracy for those measurements will be reported as determined using the actual data.

### 5.2.1 Method 0023A PCDD/PCDF and PCB Sampling Precision and Accuracy

A SW-846 Method 0023A will be used to sample the stack gas for emissions of PCDD/PCDFs. Prior to use in the field, all of the XAD-2 resin traps for use in the Method 0023A sampling train are spiked with isotopically-labeled PCDD/PCDF sampling surrogate compounds as noted in Table 5-1. Two of the XAD-2 resin traps prepared for this project will also have PCDD/PCDF matrix spikes applied and are retained

by the laboratory. These two XAD resin traps are then included with the field samples when they are returned to the laboratory for analysis. The recoveries of the PCDD/PCDF isotopically-labeled sampling surrogates and matrix spike compounds from these two retained XAD resin trap samples provide an accuracy assessment of the Method 0023A analytical methodology. The recovery of the isotopically-labeled sampling surrogates spiked onto every field-use XAD resin sample provides a field through analysis accuracy assessment of the Method 0023A methodology.

The recoveries of the internal standard, alternate standard, and recovery standard compounds applied during the preparation and analysis of the Method 0023A sampling train components provide accuracy and precision measurements for the sample preparation and analysis steps. Table 5-1 notes the RPD and RSD values for the Method 0023A PCDD/PCDF analyses.

### 5.2.2 Installed CO and $O_2$ CEMS Precision and Accuracy

The precision of the installed carbon monoxide and oxygen continuous emissions monitoring system (CEMS) analyzers will be assessed using the recommended calibration gases in accordance with 40 CFR 60, Appendix B, Specification 4B. Precision will be assessed using the following equation:

$$\text{Precision}(\%drift) = \left( \frac{R_f - R_i}{Span} \right) x100$$

*where:*

$R_f$ = Final monitor response at end of the test run

$R_i$ = Initial monitor response at start of the test run

Span = Maximum range of the analyzer.

The accuracy of all CEMS analyzers will be evaluated by the measurement of percent accuracy as defined by the equation below:

$$\text{Accuracy}(\%) = \left( \frac{R_a - R_c}{Span} \right) x100$$

where:

$R_a$ = Analyzer indicated concentration of the calibration gas

$R_c$ = Certified concentration of the calibration gas

Span = Maximum range of the analyzer.

The accuracy of oxygen and carbon monoxide CEMS analyzers will be evaluated in accordance with the CMS/CEMS Performance Evaluation Test Plan conducted prior to the test. The CEMS Performance Evaluation Test will include calibration drift tests, response time tests, calibration error tests, and relative accuracy tests per the 40 CFR 60, Appendix B, Specification 4B. During the test, the oxygen and carbon monoxide CEMS analyzers will be calibrated daily before the start of testing.

### 5.2.3 Temporary HC CEMS Precision and Accuracy

A temporary hydrocarbon (HC) analyzer will be used to demonstrate compliance with HWC MACT HC (or THC) emissions standard [40 CFR 63. 1203(a)(5)(ii) and 63.1218(a)(5)(ii)], as allowed by 40 CFR 63.1206(b)(6). The temporary HC CEM will be calibrated and operated in accordance with 40 CFR 60, Appendix A, Method 25A. The precision and accuracy of the temporary HC analyzer will be assessed during the test using the recommended calibration gases in accordance with the procedures of Method 25A. During the test, the HC analyzers will be calibrated before, at the midpoint, and at the end of each test run.

## 5.3 DETECTION LIMITS AND REPORTING

How the detection limits will be used in data reduction and reporting is described in Section 11.0.

For isotope dilution organic analysis methods, the non-detects for the isotope dilution methods will be determined using the SW-846 Method 8290 definition of an estimated detection limit (EDL) without the use of empirical factors or other mathematical manipulations specific to the laboratory. The analytical report of any target analyte that is non-detect shall include the EDL.

Each 2,3,7,8-chlorinated dioxin and furan congener, and tetra- through octa- congener group has a sample-specific EDL. When Method 0023A stack gas samples for dioxins/furans are non-detect for a target dioxin/furan, a sample-specific EDL is calculated for that analyte. This is done by determining the high resolution gas chromatograph/high resolution mass spectrometry (HRGC/HRMS) peak height of the noise or interferent in the expected region of the analyte signal. This value is typically multiplied by a factor of 2.5. The resulting signal response value is then reported as the EDL.

## 5.4 COMPLETENESS

Data completeness represents the valid data collected from the total number of valid tests conducted. Samples resulting from test runs that are judged invalid based on field performance indicators or aborted runs will not be submitted to the laboratory for analysis. Because the possibility exists that a sample may

be lost or broken, the data from each individual analytical parameter may not be complete for all test runs. The impact of any occurrence of sample loss will be assessed with regard to the objective of obtaining valid runs and will be discussed in the final test report. The completeness objective of this test program is to generate sufficient data for the regulatory agencies to judge the performance of the system.

## 5.5 REPRESENTATIVENESS AND COMPARABILITY

The sampling procedures chosen for the test program are, wherever possible, approved EPA sampling methods that are typically employed on thermal treatment system tests. The use of standard sampling methods affirms sample consistency and representativeness.

Use of standard, approved sampling and analysis methods, standardized data reduction procedures, and QC samples will provide data that is technically defensible and is comparable from test run to test run, test condition to test condition.

**Table 5-1. Test Analytical Data Quality Objectives**

| Parameter | QC type | Precision | Accuracy |
|---|---|---|---|
| **Method 0023A Sampling Train (PCDD/PCDFs)** | | | |
| Method 0023A PCDD/PCDFs | Spiked Resin Blanks | ≤ 25% RPD | 75-125% |
| Method 0023A PCDD/PCDFs | PCDD/PCDF $C^{13}$ Labeled Sampling Surrogate Spike | ≤ 35% RSD | Note 1 |
| | PCDD/PCDF $C^{13}$ Isotope Dilution Internal Standard Spikes | --- | Note 2 |
| | EPA Audit Sample | --- | 50-150% |
| **Continuous Emissions Monitors** | | | |
| Carbon Monoxide | Performance Specification 4B | ± 3% of Span | ± 5% of Span |
| Oxygen | Performance Specification 4B | ± 0.5% Oxygen | ± 0.5% Oxygen |
| Total Hydrocarbons | EPA Method 25A | ± 3% of Span | ± 5% of Certified Calibration Gas |
| **Orsat Analyses** | | | |
| Carbon Dioxide | Certified Calibration Gas | --- | ± 0.2% $CO_2$ |
| Oxygen | Ambient Air | --- | 20.9% ± 0.5% |

**Note 1: Method 0023A PCDD/PCDF Sampling Surrogate Compound Recoveries**

| Compound | Target Recovery |
|---|---|
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | 70 – 130% |
| $^{13}C_{12}$-2,3,4,7,8-PeCDF | 70 – 130% |
| $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | 70 – 130% |
| $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | 70 – 130% |
| $^{37}Cl_4$-2,3,7,8-TCDD | 70 – 130% |

**Note 2: Method 0023A PCDD/PCDF Isotope Dilution Internal Standard Compound Recoveries**

| Compound | Target Recovery |
|---|---|
| $^{13}C_{12}$-2,3,7,8-TCDD | 40 – 130% |
| $^{13}C_{12}$-2,3,7,8-TCDF | 40 – 130% |
| $^{13}C_{12}$-1,2,3,7,8-PeCDD | 40 – 130% |
| $^{13}C_{12}$-1,2,3,7,8-PeCDF | 40 – 130% |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | 40 – 130% |
| $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | 40 – 130% |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | 25 – 130% |
| $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | 25 – 130% |

# 6.0 SAMPLING AND MONITORING PROCEDURES

## 6.1 GENERAL

The objective of this test program is the collection of representative stack gas samples that demonstrate ongoing compliance of NCIN-1 and NCIN-2 with the HWC MACT emissions standard for PCDD/PCDFs. To meet this objective requires minimizing the potential sources of sample contamination or bias imparted to the samples by the sampling equipment, ambient conditions, handling, and preservation. The test program samples will be collected using the methods summarized in Table 6-1. The total numbers of field samples expected to be generated during NCIN-1 and NCIN-2 testing are also summarized Table 6-1.

Guidelines followed to determine sampling equipment to be used, sampling points, and the frequency at which samples are to be taken are presented in the test plans, and are incorporated here by reference. The reference sources for the standard sampling method references include:

- Appendix A to 40 CFR 60, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60 (EPA)
- Test Methods for Evaluating Solid Waste, SW-846, Third Edition, 1986 and updates (SW-846).

The specifics of the sampling and analytical methodologies followed during the course of test execution by the selected sampling and analytical contractors include the referenced nationally recognized methods (ASTM, SW846, 40 CFR 60 Appendix A, etc.) and/or the contractor-specific standard operating procedures (SOPs) as recognized by LELAP.

All stack sampling equipment and glassware will be prepared prior to the test according to the method specifications. Following each test run, the samples will be recovered from the sampling trains. The sample recovery procedures include prescribed rinses of the trains, which serve a dual purpose of sample recovery and decontamination of the train in preparation for the next run. Rinses that are not included in the sample recovery will be placed into a waste solvent container and disposed of by Hexion.

## 6.2 FIELD SAMPLING METHODS

### 6.2.1 Stack Gas Samples

During the test program, the Stack Sampling Coordinator and the Sample Recovery Technician are responsible for monitoring the sampling team's adherence to the standard stack sampling procedures,

especially sampling train preparation; leak checks and recoveries (including blank trains); and reagent, field, and trip blanks. The Stack Sampling Coordinator is responsible for operation and recovery of the stack sampling equipment and stack gas samples. The Sample Recovery Technician is responsible for preparing the stack gas and process samples for shipment to the laboratory. Sampling train calibration procedures are discussed in Section 8.0.

EPA Methods 1 and 2 will be used to determine the number and location of sampling traverse of isokinetic sampling locations. Documentation of Methods 1 and 2 will be included in the stack sampling report.

During each test run, Tedlar® bag samples of stack gas will be collected for determining the stack gas carbon dioxide and oxygen concentrations using EPA Method 3 (Orsat Analysis). The stack gas carbon dioxide and oxygen concentrations are used to determine the stack gas molecular weight. The Tedlar® bag samples may be collected from the exhaust of one of the isokinetic sampling trains or using a separate impinger and vacuum pump setup. Certified calibration gases containing at least 5 percent volume carbon dioxide and/or at least 5 percent volume oxygen may be used as reference standards for the Orsat apparatus. Ambient air may be used as a reference standard for the oxygen portion of the Orsat apparatus.

Stack gas moisture content will be determined for each isokinetic sampling train via EPA Method 4 (sampling train moisture gain). Isokinetic sampling train silica gel impingers will be filled with fresh, dry indicating silica gel at the beginning of the test program. During the sampling train recovery process, and subsequent test runs, each indicating silica gel impinger will be inspected prior to reuse to verify that sufficient capacity remains for moisture absorption during the next test run. Silica gel more than 50% utilized will be discarded and the impinger recharged with fresh dry indicating silica gel.

#### 6.2.1.1 Method 0023A for PCDD/PCDFs

A Method 0023A sampling train will be used to sample stack gas dioxin and furans (PCDD/PCDFs). The Method 0023A sampling train will be analyzed for PCDD/PCDFs via Method 8290 (HRGC/HRMS).

At 40 CFR 63.1208(b)(1)(iii), the HWC MACT rule requires that the Method 0023A sampling train be operated for a minimum of 180 minutes (3 hours) to sample a minimum of 2.5 dry standard cubic meters of stack gas during each sampling run so that any PCDD/PCDF congener that is non-detect may be counted as zero in determining compliance.

Prior to each test run, the Method 0023A sampling train will be assembled and leak checked. At the end of each run, the sampling train will be disassembled and all train samples collected.

#### 6.2.1.1.1 Method 0023A Recovery

The general procedures for the Method 0023A sample recovery are:

- Particulate Filter -- The particulate filter is removed from its holder and placed into its original petri dish (Container No. 1) which is sealed with Teflon tape and placed in a plastic bag.
- Front Half Acetone/Methylene Chloride Rinses -- The internal surfaces of the nozzle, probe, front half of the filter holder, and any connecting tubing or glassware are brushed and rinsed three times each with acetone, and then rinsed three times with methylene chloride. The acetone and methylene chloride front-half rinses are collected in a single glass sample bottle (Container No.2). The final liquid level is marked on the sample container.
- Front Half Toluene Rinse -- The internal surfaces of the nozzle, probe, front half of the filter holder, and any connecting tubing or glassware are rinsed two times with toluene. The front half toluene rinses are collected in a glass sample bottle (Container No. 2A). The final liquid level is marked on the sample container.
- XAD-2 Adsorbent Resin Trap -- The XAD-2 adsorbent resin trap is removed from the train, and both ends are capped. The trap is then labeled, covered with aluminum foil, sealed in a plastic bag and stored on ice in an insulated cold chest (Container No. 3).
- Back Half Acetone/Methylene Chloride Rinses -- The back half of the filter holder, the filter support, transfer line (if used), and condenser (if separate from trap) are rinsed three times with acetone followed by two rinses with methylene chloride. The acetone and methylene chloride back-half rinses are placed into a single glass sample bottle (Container No. 4). The final liquid level is marked on the sample container.
- Back Half Toluene Rinse -- The back half of the filter holder, the filter support, transfer line (if used), and condenser (if separate from trap) are rinsed two times with toluene. The back half toluene rinses are placed into a glass sample bottle (Container No. 4A). The final liquid level is marked on the sample container.
- Impinger Liquids -- The impinger contents are measured to the nearest milliliter or weighed to the nearest 0.5 g for moisture gain determination, which is recorded on the sample recovery sheet. The impinger liquids may then be discarded.
- Silica Gel -- The silica gel contents of the last impinger are weighed to the nearest 0.5 g. The color and condition of the silica gel is noted on the sample recovery sheet. The silica gel may be discarded or recovered for reuse.

#### 6.2.1.1.2 Method 0023A Preparation for Analysis

Method 0023A requires the sampling train front-half and back-half components to be prepared and analyzed as two separate fractions. Surrogates compounds discussed below are applied to each fraction:

- Sampling Surrogate Spikes – Isotopically labeled compounds spiked directly on the XAD-2 resin in the laboratory during XAD tube preparation prior to stack sampling use. The recovery of these compounds provides a comprehensive accuracy indication (stack to final analysis) of the PCDD/PCDFs found using the Method 0023A sampling method. Per Section 7.3.3 of Method 0023A, these same PCDD/PCDF surrogates are added to the Method 0023A particulate filter after sampling just prior to Soxhlet extraction.

- Isotope Dilution Internal Standard Spikes - Isotopically labeled PCDD/PCDF compounds applied to the sample just prior to the Soxhlet extraction. The recoveries of these compounds reflect the overall relative accuracy of the sample handling preparation and analysis by the laboratory.
- Alternate Standard Spike-An isotopically labeled compound may be applied to the sample after Soxhlet extraction and before extract cleanup. The recovery of this compound reflects the impacts on the sample of the extract cleanup step of sample preparation.
- PCDD/PCDF Recovery Standards- Isotopically labeled compounds applied to the Soxhlet extracts just before GC/MS analysis. These compounds are providing the relative response factors, which are used to calculate analyte concentrations.

## 6.3 FIELD QUALITY CONTROL SAMPLES

Field QC samples will be collected during the test to provide an indication of quality assurance for the test samples. The field QC samples include:

- Reagent blanks for the Method 0023A sampling train
- A blank train for Method 0023A.

Table 6-1 includes the field QC samples that will be collected.

### 6.3.1 Spiked Resin Blanks

Two XAD-2 resin traps prepared for the Method 0023A PCDD/PCDF sampling train will be spiked with a sampling surrogate and matrix spike compounds. These samples will be extracted and analyzed for PCDD/PCDFs to demonstrate the resin is free of background contamination, and to confirm that efficient surrogate recoveries are achievable.

### 6.3.2 Blank Trains and Reagent Blanks

Blank train samples are the samples recovered from sampling trains that have been assembled and charged with all the required chemical reagents and collection media in the same manner as the sampling trains used to sample the stack gases. The sampling trains are leak checked and heated to temperature in a location near the stack. The sampling train remains sealed at the stack location for a period equivalent to the length of time the corresponding sampling train is operated during the test run. The blank train is then recovered in the same way that actual stack gas sampling trains are recovered. The recovered blank train components are labeled as blank train samples and submitted for analysis with the actual stack gas train samples. The results of the blank train samples provide an indication of possible contamination introduced to the samples by reagents, glassware, sampling environment, and sampling recovery. The blank train samples are summarized in Table 6-1.

Reagent blanks are samples of the reagent source solvents, solutions, and other media used in stack sampling. Reagent blank samples are summarized in Table 6-1.

## Table 6-1. Sample Collection Locations, Equipment, and Methods

| Sample Name | Sampling Reference Method | Sample Container | Analysis | General Procedure/Frequency | NCIN-1 Test 1 | NCIN-2 Test | Field QC | Total Field Samples |
|---|---|---|---|---|---|---|---|---|
| Method 0023A Particulate Filter | SW846 Method 0023A | Petri Dish | PCDD/PCDFs | Collect integrated sample for PCDD/PCDFs during each test run. Sample for a minimum 180 minutes to sample a minimum of 3.0 dry standard cubic meters of stack gas. | 3 | 3 | 1-blank train | 7 |
| Method 0023A Front Half Acetone and Methylene | | 500 mL glass sample bottle | PCDD/PCDFs | | 3 | 3 | 1-blank train | 7 |
| Method 0023A Front Half Toluene Rinses | | 250 mL glass sample bottle | PCDD/PCDFs | | 3 | 3 | 1-blank train | 7 |
| Method 0023A XAD-2 Resin | | Resin Trap | PCDD/PCDFs | | 3 | 3 | 1-blank train | 7 |
| Method 0023A Back Half Acetone and Methylene | | 500 mL glass sample bottle | PCDD/PCDFs | | 3 | 3 | 1-blank train | 7 |
| Method 0023A Back Half Toluene Rinses | | 250 mL glass sample bottle | PCDD/PCDFs | | 3 | 3 | 1-blank train | 7 |
| Method 0023A Acetone Reagent Blank | SW846 Method 0023A | 250 mL glass sample bottle | PCDD/PCDFs | Reagent Blank | -- | -- | 1 | 1 |
| Method 0023A Methylene Chloride Reagent Blank | SW846 Method 0023A | 250 mL glass sample bottle | PCDD/PCDFs | Reagent Blank | -- | -- | 1 | 1 |
| Method 0023A Toluene Reagent Blank | SW846 Method 0023A | 250 mL glass sample bottle | PCDD/PCDFs | Reagent Blank | -- | -- | 1 | 1 |
| PCDD/PCDF Audit Sample | SW846 Method 8290 | Ampoule with spiked XAD-2 Resin | PCDD/PCDFs | As provided by Agency | -- | -- | 1 | 1 |
| Carbon Dioxide and Oxygen by Orsat | EPA Methods 3 and 3A | 25-50 L Tedlar gas sample bag | Carbon Dioxide and Oxygen by Orsat | Collect two 1-hour integrated bag sample for on-site Orsat analysis. | 6 | 6 | -- | 12 |
| Total Hydrocarbons | EPA Methods 25A | Temporary Continuous Emissions Monitor | Total Hydrocarbons | Continuous during each test run | -- | -- | -- | -- |
| Carbon Monoxide | EPA Method 10; Performance Specification 4B | Installed Continuous Emissions Monitor | Carbon Monoxide | Continuous during each test run | -- | -- | -- | -- |
| Oxygen | EPA Method 3A; Performance Specification 4B | Installed Continuous Emissions Monitor | Oxygen | Continuous during each test run | -- | -- | -- | -- |
| **TOTAL SAMPLES** | | | | | | | | **58** |

# 7.0 SAMPLE HANDLING, TRACEABILITY, AND HOLDING TIMES

## 7.1 SAMPLE CUSTODY AND SECURITY

A sample will be considered to be in the custody of a person if it is in his or her possession, in his or her sight, or secured by that person in an approved location accessible only to authorized personnel.

During the test, once the samples are transferred from Stack Sampling Technicians to the Sample Recovery Technician, sample custody becomes the responsibility of the Sample Recovery Technician until the samples arrive at the analytical laboratory. When overnight couriers are utilized, the air bill will serve to document the transfer of custody from the Sample Recovery Technician to the courier. The courier's air bill becomes part of the chain of custody (COC) record. Upon transfer of the samples from the courier to the analytical laboratory, sample custody will be maintained by the analytical laboratory performing the analyses.

Samples will be kept on ice ($4\pm2^{o}C$) and shipped to the analytical laboratory in sealed, insulated shipping containers. All ice used for shipping samples shall be double bagged in Ziplock® bags to prevent leakage of water during shipping. "Blue ice" and "dry ice" may also be used to chill samples.

## 7.2 SAMPLE IDENTIFICATION

Refer to Figure 7-1. The Laboratory Analysis Coordinator will prepare the master sample list for the test program. Alpha-numeric sample numbers will be assigned to every sample. The sample numbers are used to track individual samples from collection through analysis. From the master sample list, the analytical laboratory will prepare the pre-labeled EPA Class III clean sample containers for field use, and the XAD-2 resins according to the specifications of the methods as described in the test plan. These items will be shipped to the test site in sealed transport containers. The stack sampling contractor will provide the sampling reagents for field use.

## 7.3 STACK SAMPLE COLLECTION FORMS

While stack sampling is being performed, the sampling technician will complete a stack sampling record. An example stack field sampling record for iso-kinetic sampling is presented as Figure 7-2. The stack sampling record will be completed in its entirety for every sampling train. This will provide information necessary to perform the emissions calculations. The Stack Sampling Technician shall provide the completed stack sampling record to the Stack Sampling Coordinator at the completion of each sampling run.

## 7.4 SAMPLE LABELING

An example sample label format is presented in Figure 7-3. Each sample container will be labeled to show the source of the sample as Hexion; the project identification; sampler's initials; laboratory to which the sample will be shipped; the unique alphanumeric sample number; date and time; sample description; test number; and run number. If a single sample requires multiple containers, the number of the container and the total number of containers will be noted on the label. Project samples will be tracked via the assigned unique alphanumeric sample numbers. The sample number will appear on the sample label, the request for analysis (RFA), and the chain of custody (COC).

## 7.5 SAMPLE COLLECTION CHECKLIST

The master sample list identifies every sample by the assigned unique alphanumeric sample numbers (Refer to Figure 7-1), and the corresponding analytical test(s) required. As field samples are acquired, the samples will be checked off against the master list to ensure that all of the appropriate samples have been collected.

## 7.6 REQUEST FOR ANALYSIS/CHAIN OF CUSTODY

Collected samples will be shipped from the site to the laboratory in sealed containers with COC and RFA forms. Example RFA and COC forms are presented as Figures 7-4 and 7-5 respectively. The Stack Sampling Technician and Sample Recovery Technician will complete the COC and RFA forms for every sample. Some samples may consist of several sub-samples. Each individual component of the sample will be listed separately on the RFA/COC with its own unique alphanumeric sample identification number. The samples will be preserved as needed, and will remain in the possession of the Sample Recovery Technician. The Sample Recovery Technician will secure the samples in a location accessible only to authorized personnel until custody is transferred to a courier for delivery to the laboratory.

## 7.7 SAMPLE SHIPMENT

Field samples may be transported directly to the analytical laboratory by the sampling contractor. If the samples are shipped via overnight courier, e.g. Federal Express, an individual trained in Federal Department of Transportation (DOT) and International Air Transport Association (IATA) regulations will package the samples to assure compliance with the applicable portions of these regulations.

Prior to shipping any samples, the Sample Recovery Technician will verify the condition of the samples: sample temperatures for organic analyses samples, condition of all containers, level of sample within all containers (to be marked on the outside of the container), and type of packing material used. RFA/COCs will be checked to verify there is a RFA and COC for every sample being shipped. Before shipping the

samples from the site, the Sample Recovery Technician shall make and retain a photocopy set of all the RFA/COCs.

### 7.8 SAMPLE DELIVERY

Upon receipt of samples at the laboratory, the receiver will accept custody for the shipment by an exchange of signatures with the delivering agent. The shipping containers will be opened by the Laboratory Analysis Coordinator or his designee and inspected. The container contents will verified against the accompanying RFA/COC. Any damage to the contents of the shipping container or deviations from the original shipment documents will be noted on the COC. The Laboratory Analysis Coordinator or designee will, immediately upon opening the sample packaging, measure the temperature of the samples using a thermometer. This temperature will be recorded on the COCs and any applicable laboratory documentation (sample receipt log).

Transfer of custody to and within the analytical laboratory is addressed in the Laboratory's QA Manual. Upon completion of analysis, samples will be maintained at the laboratory under COC until they are released for proper disposal.

### 7.9 SAMPLE PRESERVATION

Table 7-1 shows the appropriate containers, preservation, and holding times for all samples to be collected during the test. XAD-2 traps and other train sample components for the Method 0023A PCDD/PCDF sampling trains will be preserved after sampling by placing them in a cooler on double bagged ice or blue ice.

**Table 7-1. Sample Containers, Preservation, and Holding Times**

| Parameter | Sample Name/Matrix | Sample Containers | Preservation | Maximum Holding Time |
|---|---|---|---|---|
| Stack Gas PCDD/PCDFs, | Stack Gas Method 0023A filter | Glass petri dish | Chill to 4°C | 30 days until extraction, 45 days after extraction |
| | Stack Gas Method 0023A sorbent tube | Standard cartridge wrapped in aluminum foil and sealed in plastic bag | Chill to 4°C | 30 days until extraction, 45 days after extraction |
| | Stack Gas Method 0023A solvents | Amber glass Boston round bottles with Teflon-lined caps | Chill to 4°C | 30 days until extraction, 45 days after extraction |

Table Notes:

[a] Reference: Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration, EPA/625/6-89/023, January, 1990 and promulgated method.

Hexion PCDD/PCDF Confirmatory Test
Norco, LA
Focus Project No. 060804

Test America, Knoxville, TN
March 2, 2009

## PCDD/PCDF CONFIRMATORY TEST MASTER SAMPLE LIST

| Field Sample No. | Test No. | Run No. | RFA/COC No. | Sample Source | Sample Description | Sample Container | Analytical Parameters | Analytical Laboratory | QC Analysis |
|---|---|---|---|---|---|---|---|---|---|
| K- 3010 | 1 | 1 | 003 | Method 0023A Train | Particulate Filter | Petri dish | Dioxins and Furans | TA, Knoxville | |
| K- 3011 | 1 | 1 | 007 | Method 0023A Train | XAD-2 Resin Tube | XAD-2 Resin Tube | Dioxins and Furans | TA, Knoxville | |
| K- 3012 | 1 | 1 | 007 | Method 0023A Train | Front Half Filter Holder & Probe Rinses | 500 mL amber bottle | Dioxins and Furans | TA, Knoxville | |
| K- 3013 | 1 | 1 | 007 | Method 0023A Train | Back Half Filter Holder & Probe Rinses | 500 mL amber bottle | Dioxins and Furans | TA, Knoxville | |
| K- 3014 | 1 | 1 | 007 | Method 0023A Train | Front Half Toluene Probe Rinse | 500 mL amber bottle | Dioxins and Furans | TA, Knoxville | |
| K- 3015 | 1 | 1 | 007 | Method 0023A Train | Back Half Toluene Probe Rinse | 500 mL amber bottle | Dioxins and Furans | TA, Knoxville | |
| K- 3016 | 1 | 1 | 007 | Method 0023A Train | Blank Train Particulate Filter | Petri dish | Dioxins and Furans | TA, Knoxville | BT |
| K- 3017 | 1 | 1 | 007 | Method 0023A Blank Train | Blank Train XAD-2 Resin Tube | XAD-2 Resin Tube | Dioxins and Furans | TA, Knoxville | BT |
| K- 3018 | 1 | 1 | 007 | Method 0023A Blank Train | Blank Train FH Filter Holder & Probe Rir | 500 mL amber bottle | Dioxins and Furans | TA, Knoxville | BT |
| K- 3019 | 1 | 1 | 007 | Method 0023A Blank Train | Blank Train BH Filter Holder & Probe Ri | 500 mL amber bottle | Dioxins and Furans | TA, Knoxville | BT |
| K- 3020 | 1 | 1 | 007 | Method 0023A Blank Train | Blank Train FH Toluene Probe Rinse | 500 mL amber bottle | Dioxins and Furans | TA, Knoxville | BT |
| K- 3021 | 1 | 1 | 007 | Method 0023A Blank Train | Blank Train BH Toluene Probe Rinse | 500 mL amber bottle | Dioxins and Furans | TA, Knoxville | BT |

**Figure 7-1. Example Master Sample List**

**Isokinetic Stack Sample Data Collection Sheet**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Contract No. | 116029 | Method: | M0023A (Dioxin) | | | Page 1 of 1 | |
| Facility | Hexion, Norco, LA | Initial Leak Rate ($ft^3$ @ in.Hg) | <0.010 @ 10" | | | Operator | MJK |
| Source | NCIN-1 | Final Leak Rate ($ft^3$ @ in.Hg) | <0.010 @ 10" | | | Pitot No. | K-004 |
| Date | 18-Mar-09 | Start Time | 0800 | Meter No. | 1442 | PTCF | 0.84 |
| Condition No. | 3 | End Time | 1015 | DGMCF | 1.004 | Init. Pitot Leak Check | 0.0 |
| Run No. | 1 | Duration (min) | 120 | H @ | 1.645 | Final Pitot Leak Check | 0.0 |
| Stat. Press. (in. $H_2O$) | -0.94 | Bar. Press. (in.Hg) | 29.73 | Nozzle Dia.( ") | 0.251 | Kf | 2.3 |

| Point | Time | Volume | P | H | Temperatures (°F) | | | | | | | Vacuum |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| No. | (24 Hr) | ($ft^3$) | (in. $H_2O$) | (in. $H_2O$) | Flue Gas | Probe | Filter | Impingers | Meter in | Meter Out | Cond. Exit | (in. Hg) |
| A-1 | 0800 | 985.95 | 0.78 | 1.79 | 294 | 225 | 258 | 68 | 103 | 105 | 58 | 6.0 |
| A-1 | 0808 | 991.03 | 0.78 | 1.79 | 294 | 260 | 257 | 67 | 105 | 104 | 58 | 6.1 |
| A-2 | 0816 | 996.10 | 0.74 | 0.17 | 294 | 257 | 261 | 66 | 106 | 104 | 58 | 6.1 |
| A-2 | 0824 | 1000.94 | 0.74 | 1.7 | 294 | 261 | 258 | 66 | 107 | 104 | 59 | 6.1 |
| A-3 | 0832 | 1005.33 | 0.68 | 1.56 | 292 | 258 | 257 | 64 | 108 | 104 | 60 | 6.1 |
| A-3 | 0840 | 1010.10 | 0.68 | 1.56 | 292 | 257 | 258 | 63 | 109 | 104 | 62 | 6.1 |
| A-4 | 0848 | 1015.50 | 0.77 | 1.77 | 292 | 258 | 258 | 67 | 104 | 104 | 56 | 6.1 |
| A-4 | 0856 | 1021.09 | 0.77 | 1.77 | 292 | 258 | 256 | 67 | 109 | 103 | 56 | 6.2 |
| A-5 | 0904 | 1026.48 | 0.77 | 1.7 | 293 | 258 | 259 | 61 | 110 | 104 | 56 | 6.2 |
| A-5 | 0912 | 1031.94 | 0.77 | 1.7 | 289 | 256 | 258 | 61 | 108 | 104 | 57 | 6.2 |
| A-6 | 0920 | 1036.94 | 0.74 | 1.43 | 285 | 259 | 255 | 62 | 106 | 105 | 58 | 6.2 |
| A-6 | 0928 | 1041.77 | 0.74 | 1.43 | 290 | 258 | 255 | 62 | 105 | 103 | 58 | 6.3 |
| STOP | 0936 | 1046.09 | | | | | | | | | | |
| B-1 | 0950 | 1046.39 | 0.62 | 1.77 | 292 | 255 | 259 | 66 | 96 | 95 | 58 | 6.2 |
| B-1 | 0958 | 1051.32 | 0.62 | 1.77 | 292 | 259 | 257 | 65 | 96 | 95 | 59 | 6.2 |
| B-2 | 1006 | 1056.66 | 0.77 | 1.43 | 293 | 257 | 257 | 62 | 99 | 95 | 61 | 6.3 |
| B-2 | 1014 | 1061.49 | 0.77 | 1.29 | 289 | 257 | 258 | 62 | 102 | 95 | 56 | 6.4 |
| B-3 | 1022 | 1066.13 | 0.62 | 1.04 | 285 | 258 | 257 | 62 | 104 | 97 | 56 | 6.4 |
| B-3 | 1030 | 1070.33 | 0.56 | 1.1 | 290 | 257 | 253 | 61 | 106 | 99 | 56 | 6.4 |
| B-4 | 1038 | 1074.91 | 0.45 | 1.5 | 296 | 257 | 257 | 63 | 109 | 100 | 57 | 6.4 |
| B-4 | 1046 | 1075.09 | 0.56 | 1.5 | 296 | 253 | 258 | 64 | 111 | 101 | 58 | 6.5 |
| B-5 | 1054 | 1084.31 | 0.45 | 0.99 | 296 | 257 | 259 | 61 | 112 | 103 | 58 | 6.5 |
| B-5 | 1102 | 1088.53 | 0.44 | 0.94 | 297 | 258 | 258 | 60 | 112 | 104 | 58 | 6.5 |
| B-6 | 1110 | 1092.66 | 0.65 | 0.85 | 297 | 259 | 258 | 61 | 113 | 105 | 58 | 6.5 |
| B-6 | 1118 | 1096.44 | 0.65 | 0.85 | 294 | 258 | 257 | 61 | 112 | 105 | 59 | 6.5 |
| END | 1126 | 1100.32 | | | | | | | | | | |
| Comments: | | | | | | | | | | | | |

**Figure 7-2. Example Stack Sampling Record**

**Hexion NCIN-1 PCDD/PCDF Confirmatory Test**
**Norco, LA**
**Focus Project No. 060804**

| | | | |
|---|---|---|---|
| **Sample Type:** | Method 0023A Filter | **Sample No.:** | **K-3010** |
| **Test No.** ___1___ | **Run No.** ___1___ | **Container(s):** | ___ of ___ |
| **Analysis Required:** | Dioxins and furans | | |
| **Analysis Laboratory:** | Test America, Knoxville, TN | | |
| **Date:** | 18-Mar-09 | **Initials:** | MJK |
| **Time:** | 1415 | **Preservation:** | NA |

**Figure 7-3. Example Sample Label Format**

## Request for Analysis/Chain of Custody No. 007

**PCDD/PCDF Hexion Confirmatory est**

**Norco, LA**
**Focus Project No. 060804**

**Test America-Knoxville Lot No. _H-XXXXXXXX_____**
**Test America-Knoxville Project No. ___XXXXXXX____**

| | |
|---|---|
| **Project Description:** | Dioxin Test Program |
| **Client Project No.:** | 060804 |
| **Test America Project No:** | XXXXXXX |
| **Client Project Mgr:** | Chris McBride |
| | 865-694-7517 x3041 |
| **Test America Contact:** | Patti Carswell |
| | 865-291-3010 |
| **Test America Project Mgr:** | Dr. William C. Anderson |
| | 865-291-3080 |

**Laboratory Deliverable Requirements**

Analytical Due Date: 21 days from lab receipt

Data Package Due Date: 30 days from lab receipt

**Analytical Testing QC Requirements:**
BT- Blank train

**Laboratory Destination:**

Laboratory Destination
Test America-Knoxville
5815 Middlebrook Pike
Knoxville, TN 37921
(865)-291-3000

**Courier:** Hand deliver

**Project Deliverables:**

| **Holding Time Requirements:** | |
|---|---|
| Dioxins | 30 days to analysis |
| Dioxins | 45 days after extraction to analysis |

| Field Sample No. | Test No. | Run No. | Sample Collection Date/ Time | Sample Container | Sample Description | Analysis Specifications | Project QC Requirements |
|---|---|---|---|---|---|---|---|
| K- 3010 | 1 | 1 | 18-Mar-09 1415 | Petri dish | Method 0023A Train Particulate Filter | Combine this sample with the corresponding front half sovlent rinses, Sample No. K-3013 and K-3014. Perform Method 0023A extraction and analyze via Method 8290 for Dioxins and Furans. | |
| K- 3011 | 1 | 1 | 18-Mar-09 1415 | 500 mL amber bottle | Method 0023A Train Front half of filter holder & probe solvent rinses probe rinse | Combine this sample with the corresponding filter and front half toluene rinse, Sample No. K-3012 and K-3014. Perform Method 0023A extraction and analyze via Method 8290 for Dioxins and Furans. | |
| K- 3012 | 1 | 1 | 18-Mar-09 1415 | 500 mL amber bottle | Method 0023A Train Front half of toluene probe solvent rinses probe rinse | Combine this sample with the corresponding filter and front half sovlent rinse, Sample No. K-3012 and K-3013. Perform Method 0023A extraction and analyze via Method 8290 for Dioxins and Furans. | |

**Figure 7-4. Example Request for Analysis**

**Request for Analysis/Chain of Custody No. 007**
**Hexion Dioxin Confirmatory Test**
**Norco, LA**
**Focus Project No. 060804**

**Sample Receipt Log and Condition of the Samples Upon Receipt**

| Please fill in the following information: | | Comments |
|---|---|---|
| | | (Please write "NONE" if no comment is applicable.) |
| (1) | Record the identities of any samples that were listed on the Request for Analysis form but were not found in the sample shipment | |
| (2) | Record the sample shipping cooler temperature of all coolers transporting samples listed on the Request for Analysis form. | |
| (3) | Record any apparent sample loss or breakage. | |
| (4) | Record any unidentified samples transported with this shipment of samples. | |
| (5) | Indicate if all samples were received according to the project's required specifications (i.e, no non-conformances). | |

**Custody Transfer**

| | Name | Company | Date/Time |
|---|---|---|---|
| Relinquished by: | | | |
| Accepted by: | | | |
| Relinquished by: | | | |
| Accepted by: | | | |
| Relinquished by: | | | |
| Accepted by: | | | |

**Figure 7-5. Example Chain of Custody**

# 8.0 SPECIFIC CALIBRATION PROCEDURES AND FREQUENCY

The objective of this section is to assure that process instrumentation, gas sampling equipment, and analytical instruments are performing properly before conducting the test and analyzing samples. Equipment and instruments used to generate data for determining compliance with performance requirements or to establish quantitative allowable operating limits will be calibrated according to the manufacturer's instructions, prior to and/or during the test as necessary.

The calibration procedures are separated into groups according to the personnel who will perform them. Hexion operations personnel will calibrate the process instruments. Stack sampling equipment will be calibrated by the stack sampling contractor and analytical instruments will be calibrated by the contracted laboratory personnel. The calibration procedures for process instrumentation, stack gas sampling, and laboratory analytical instruments are described in the following subsections.

## 8.1 PROCESS INSTRUMENTATION

Within 30 days of the start of testing, the parameter continuous monitoring system (CMS) (thermocouples, flow meters, pressure transducers, etc.) will be calibrated in accordance with the CMS/CEMS Performance Evaluation Test Plan prepared in accordance with 40 CFR 63.8(e).

As required by Section 5.0 of Appendix to Subpart EEE of Part 63--Quality Assurance Procedures for Continuous Emissions Monitors Used for Hazardous Waste Combustors, the continuous emission monitoring system (CEMS) (installed CO and $O_2$ monitors) will be undergo a Relative Accuracy Test Audit (RATA) within 90 days prior to the test. The RATA will include calibration drift tests, response time tests, calibration error tests, and relative accuracy tests. During testing, the installed carbon monoxide and oxygen CEMS, the monitors will be calibrated daily before testing. The zero and span checks will be considered a verification of the data quality from these monitors.

CMS and CEMS data will be reported on 1-minute intervals and will be archived in the CMS data acquisition system.

## 8.2 STACK SAMPLING EQUIPMENT

Sampling equipment is calibrated according to the criteria specified in the reference method being employed. In addition, the guidelines set forth in the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods (EPA-600/4-77-027b) will be followed. Dry gas meters, orifices, nozzles, pitot tubes, etc. are calibrated in accordance with this

document. The range of the calibration is specified for all environmental measurements to encompass the range of probable experimental values. This approach ensures that all results are based upon interpolative analyses rather than extrapolative analyses.

Calibrations are designed to include, where practical, at least three measurement points evenly spaced over the range. This practice minimizes the probability that false assumptions of calibration linearity will be made. In addition it is common practice to select, when practical, at least one calibration value approximating the levels anticipated in the actual measurement. Typically, calibration frequency is dictated by the need to demonstrate the stability of the calibration value over the course of measurements. Calibrations are made both pre- and post-test to accomplish the demonstration of stability.

Following the test program, calibrations are checked on all relevant items of sampling equipment to ensure the validity of data collected in the field. New items for which calibration is required are calibrated before initial field use. Equipment whose calibration status may change with use or time is inspected in the field before testing begins and again upon return from each field use. When an item of equipment is found to be out of calibration, it is repaired and recalibrated or retired from service. All equipment is periodically recalibrated in full, regardless of the outcome of these regular inspections.

Data obtained during calibrations are recorded on standardized forms, which are checked for completeness and accuracy by management personnel. Data reduction and subsequent calculations are performed using standard procedures, and are computerized where appropriate. Calculations are checked at least twice for accuracy. Copies of calibration forms are included in the test or project reports.

Emissions sampling equipment requiring calibration include pitot tubes, pressure gauges, thermometers, dry gas meters, and barometers. The following sections elaborate on the calibration procedures for these specific equipment items.

### 8.2.1 Pitot Tubes

All Type S pitot tubes, whether separate or attached to a sampling probe, are inspected in accordance with the geometry standards contained in EPA Method 2.

For Type S pitot tubes with a $D_t$ between 3/16 and 3/8 inches, the pitot tube may be calibrated according to the procedure outlined in Sections 10.1.2 through 10.1.5 of Method 2 before and after the test, or a baseline (isolated tube) coefficient value of 0.84 may be assigned.

All Type S pitot tubes >3/8 inches are calibrated over an eight-point range with a wind tunnel and a calibration coefficient is calculated for each pitot tube. The acceptance limits are listed in Table 8-1.

### 8.2.2 Differential Pressure Gauges

Some meter consoles are equipped with 10-inch water column (W.C.) inclined-vertical manometers. Fluid manometers do not require calibration other than leak-checks. Manometers are leak-checked in the field prior to each test series.

### 8.2.3 Digital Temperature Indicator

One digital temperature indicator is used to determine the flue gas temperature, probe temperature, oven temperature, "train temperature" and dry gas meter temperature. The digital temperature indicator is calibrated over a seven-point range (32°F-450°F) using an ASTM mercury-in-glass thermometer as a reference. The calibration is acceptable if the agreement is within ±2% or 2°F from 50°F-180°F.

### 8.2.4 Dry Gas Meter and Orifice

A calibrated wet test meter is used to calibrate the dry gas meter and orifice. The full calibration procedure is used to obtain the calibration factor of the dry gas meter. Full calibrations are performed using a calibrated wet test meter as a reference standard.

#### 8.2.4.1 Dry Gas Meter

Each metering system receives a full calibration at the time of purchase and quarterly. Upon request, a post-test calibration can be performed after each field use. If the calibration factor deviates by less than five percent from the initial value, the test data are acceptable. If it deviates by more than 5%, the meter is recalibrated and the meter coefficient (initial or recalibrated) that yields the lowest sample volume for the test runs is used.

EPA Method 5 requires another full calibration anytime the post-test calibration check indicates that the calibration factor has changed by more than 5%. Standard practice is to recalibrate the dry gas meter quarterly and check the orifice calibration during and after each field use.

#### 8.2.4.2 Orifice

An orifice calibration factor is calculated for each of the eighteen flow settings during a full calibration. The arithmetic average of the values obtained during the calibration is used.

### 8.2.5 Barometer

Each field barometer is adjusted before each test series to agree within ±0.1 inches of a reference aneroid barometer. The reference barometer is checked against the weather station pressure value (corrected for elevation difference) reported by the National Weather Service or mercury barometer. This information is obtained via a call to the weather line or the nearest airport

## 8.3 LABORATORY ANALYTICAL EQUIPMENT

The laboratory instruments will be calibrated as specified by the appropriate method before analyzing the test samples. The laboratory instrument calibration procedures are based on instructions in the referenced analytical methods and are summarized, along with other routine quality control checks, in Table 8-2. The calibrations performed and the results will be reported as appropriate to assure the quality of data in the laboratory sample analysis report.

**Table 8-1. Sampling Equipment Calibration Requirements**

| Stack Sampling Equipment | Acceptance Criteria | Measurement Frequency | Action If Criteria Are Not Met |
|---|---|---|---|
| **Volumetric Flow Measurements** | | | |
| Type S pitot tube inspection | All dimension specifications met | Calibrate prior to test and visually inspect after each field test | Use pitot tubes that meet face opening specifications; repair or replace as required |
| Type S pitot tube calibration | ± 3% for volumetric flows >1,000 fpm<br>± 5-6% for volumetric flows >600 and <1,000 fpm | Refer to Section 10.0 of Method 2: If $D_t$ is between 0.48 and 0.95 cm (3/16 and 3/8 in.), and if $P_A$ and $P_B$ are equal and between 1.05 and 1.50 $D_t$, there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 10.1.2 through 10.1.5 of Method 2 before and after the test, or (2) a baseline (isolated tube) coefficient value of 0.84 may be assigned to the pitot tube with pitot tube inspection before and after the test. | Check for blockage. If blockage is significant, recalculate calibration coefficient. |
| Barometers | ±0.1 inches Hg (± 2.5mm Hg) of mercury-in-glass barometer | Calibrate initially versus mercury-in-glass barometer; check before and after field test | Adjust to agree with a certified barometer |
| Stack gas temperature measurement system | Capable of measuring within ± 2°F (± 1°C) of mercury-in-glass thermometer | Calibrate prior to test and after each field use | Adjust to agree with Hg bulb thermometer; construct calibration curve, correct readings |
| Pressure sensors (excludes inclined manometer) | Agree within ± 5 percent of inclined manometers | Prior to and after field use | Adjust to agree with Hg bulb thermometer; construct calibration curve, correct readings |
| Wet test meter | $Y_{mi} = Y_m \pm 0.030$ Y (before test)<br>$Y_{mf}/Y_{mi}$ is 0.95 to 1.05 (after test) | Calibration prior to test<br>Check calibration after test | Before test: Adjust until specifications are met.<br>After test: Recalculate calibration coefficient. |

**Table 8-1. Sampling Equipment Calibration Requirements**

| Stack Sampling Equipment | Acceptance Criteria | Measurement Frequency | Action If Criteria Are Not Met |
|---|---|---|---|
| Dry gas meters | $Y_i = Y \pm 0.02\ Y$ (before test)<br>$Y_{mf}/Y_{mi}$ is 0.95 to 1.05 (after test) | Calibration versus wet test meter initially, and when post-check exceeds $\Delta Y \pm 0.05$ | Before test: Repair or replace and then calibrate<br>After test: Recalculate calibration coefficient. |
| Assembled isokinetic sampling train leakage | 0.02 cfm (0.00057 $m^3$/min) at vacuum of $\geq$ 10 inches Hg (250 mm Hg) before the start of stack sampling, and $\geq$ maximum vacuum value recorded during sampling run for post sampling leak checks | Just prior to start of first sampling traverse (required); after first sampling traverse (recommended); after moving sampling train from first traverse port to second traverse port (recommended); end of second sampling traverse (required). | Before stack sampling or at post port change: Isolate and repair leak point(s); repeat leak check.<br>End of first traverse or end of second traverse: Determine leak rate; If <4% of sampling rate, correct sample volume per procedures in Section 6.3 of Method 5.<br>If >4% of sampling rate, invalid test sample; discard sample and repeat sampling run for invalid train. |
| Tedlar bags | Any water manometer displacement after 10 minutes when inflated to 2 to 4 inches (5 to 10 cm) water column pressure | Every bag | Discard bag |
| Orsat analyzer leak check | Using bag or calibration gas, bring liquid level in each pipette to the reference mark and close stopcock. Burette meniscus changes < 0.2 mL after 4 minutes. | Before and after test | Replace leaking tubing or stopcock valves, or defective hose tubing clamps. |
| Viability of Orsat analyzer solutions | Using oxygen and carbon dioxide calibration gases, conduct Orsat analyses; $\pm$0.5% of calibration standard | Before and after test | Replace spent absorbing solution with fresh solution. |
| Analytical balance (for moisture) | ± 1 mg of Class-S weights | Check with Class-S weights upon receipt and daily | Adjust or repair |

**Table 8-1. Sampling Equipment Calibration Requirements**

| Stack Sampling Equipment | Acceptance Criteria | Measurement Frequency | Action If Criteria Are Not Met |
|---|---|---|---|
| **Sampling Train Heating Systems and Thermocouples** | | | |
| Probe heating system (isokinetic sampling trains) | Capable of maintaining 248° ± 25°F (120°C ± 14°C) at a flow of 0.75 cfm (21.2 L/min) | Calibrate initially by APTD-0576(11) or use published calibration curves | Repair, or replace, and then verify the calibration |
| Probe nozzle (isokinetic sampling trains) | Average of three ID measurements of nozzle within 0.001 inches (0.0025 mm); difference between high and low 0.002 inches (0.0050 mm) | Use a micrometer to measure to nearest 0.025mm (0.001 in.); check before and after field test | Recalibrate, reshape, and sharpen when nozzle becomes nicked, dented, or corroded |
| Thermocouples (stack gas meters and final impinger) | Impinger thermocouple ± 1°C (2°F) [Method 5]; dry gas thermocouple ± 3°C (5.4°F) [Method 0010]; stack thermocouple within ± 1°C(± 2°F) of absolute temperature. | Calibrate prior to test against a mercury thermometer | Adjust; determine a correction factor or reject |

Sources:
Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods, EPA-600/R-94/038c, September 1994.
Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment, APTD-0576, U.S. EPA, Office of Air Programs, March 1972.
Construction of Isokinetic Source Sampling Equipment, APTD-0581, U.S. EPA, Office of Air Programs, April 1971.
Determination of Particulate Emissions from Stationary Sources, U.S. EPA, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60, Appendix A.

**Table 8-2. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions**

| Parameter/Method | Quality Control Check | Method of Determination | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|---|
| PCDD/PCDFs by High Resolution GC/High Resolution MS (SW846 8290 | Initial Calibration | All five high resolution concentration calibration solutions must be used for the initial calibration | Prior to sample analysis | PCDD/PCDF: The %RSD for the mean RRF from the each of the unlabeled standards must not exceed ± 20%, and those for the labeled reference compounds must not exceed ± 30%.<br>PCB: Mean RRF %RSD must be no greater than 30% for both unlabeled analytes and internal standards | Recalibrate |
| | Continuing Calibration | Midlevel standard | At the beginning and end of each 12 hour shift | PCDD/PCDF: RFs must be within ± 20% of the initial calibration mean RRF for unlabeled standards and ± 30% for labeled standards<br>PCB: RRFs must be within ±30% of the initial calibration mean RRF for all analytes (both labeled and unlabeled) | Reanalyze standard. If second analysis does not meet criteria, recalibrate and reanalyze samples or justify acceptance of sample results since the last successful check. |
| | Retention time window verification and GC column performance | Monitor retention times | Start of each 12 hour shift | PCDD/PCDF: Compliance with Section 8.2.1 of Method 8290<br>PCB: The internal standard RT must be within ± 10 seconds of the continuing calibration. | Correct according to method |
| | Method Blank | Analysis of blanks | Analyze one with each analytical batch | Results less than method detection limit | Flag data as discussed in case narrative |

## 9.0 ANALYTICAL PROCEDURES

Analytical procedures and methods are summarized in Tables 9-1. Individual analytical methods are described in detail in Appendix C of the Confirmatory Test Plan and are incorporated here by reference. The following is a list of the analytical reference methods for the procedures presented in Table 9-1:

- Test Methods for Evaluating Solid Waste, SW-846 (SW-846), Third Edition, November 1986 and Updates
- Sampling and Analysis Methods for Hazardous Waste Incineration, EPA 600/8-84-002.
- American Society for Testing and Materials (ASTM), Annual Book of ASTM Standards, Philadelphia, Pennsylvania, Annual Series
- Appendix A, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60.
- Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020.
- Guidance for Total Organics, EPA/600/R-96/036, March, 1996
- Performance Specification 4B, Appendix B, 40 CFR 60.

Table 9-2 presents the stack gas PCDD/PCDF target analytes for the Method 0023A samples and expected detection limits.

The selected analytical laboratory is LELAP qualified for each type of sample analysis delineated in this test plan. Laboratory LELAP qualifications are included as Attachment B to this QAPP. Reference sampling and analytical methods are identified throughout the test plans and this QAPP. The specifics of the sampling and analytical methodologies followed during the course of test execution by the selected sampling and analytical contractors include the referenced nationally recognized methods (ASTM, SW846, 40 CFR 60 Appendix A, etc.) and/or the contractor-specific standard operating procedures (SOPs) as recognized by the LELAP.

## Table 9-1. Summary of Analytical Procedures and Methods
## Page 1 of 2

| Analysis | Sample Matrix | Field Samples | | | Reference Preparation Method | Reference Analytical Method | QC Analysis | QC Analysis Frequency | QC Analyses | Total Analyses |
|---|---|---|---|---|---|---|---|---|---|---|
| | | NCIN-1 Test 1 | NCIN-2 Test | Field QC | | | | | | |
| PCDD/PCDFs by SW846 Method 0023A | Method 0023A front half composite; filter, and front half of filter holder and probe rinses. | 3 | 3 | 1 | Soxhlet extraction with MeCl2 (SW-846 Methods 0023A/3542) | HRGC/HRMS for PCDD/PDDFs (SW-846 Method 8290) | PCDD/PCDF isotope dilution internal standard spike | Every analysis | 7 | 7 |
| | | | | | | | PCDD/PCDF recovery standard spike | Every analysis | 7 | |
| | Method 0023A back half composite; XAD-2 resin, and back half of filter holder and condenser rinses. | 3 | 3 | 1 | Soxhlet extraction with MeCl2 (SW-846 Methods 0023A/3542) | HRGC/HRMS for PCDD/PCDFs (SW-846 Method 8290) | PCDD/PCDF pre-sampling surrogate spikes | Every XAD-2 resin tube before sampling | 7 | 7 |
| | | | | | | | PCDD/PCDF isotope dilution internal standard spike | Every analysis | 7 | |
| | | | | | | | PCDD/PCDF recovery standard spike | Every analysis | 7 | |
| | Method 0023A acetone reagent blank | -- | -- | 1 | | HRGC/HRMS for PCDD/PCDFs (SW-846 Method 8290) | PCDD/PCDF isotope dilution internal standard spike | Every analysis | 1 | 1 |
| | | | | | | | PCDD/PCDF recovery standard spike | Every sample | 1 | |
| | Method 0023A methylene chloride reagent blank | -- | -- | 1 | | HRGC/HRMS for PCDD/PDDFs (SW-846 Method 8290) | PCDD/PCDF isotope dilution internal standard spike | Every analysis | 1 | 1 |
| | | | | | | | PCDD/PCDF recovery standard spike | Every analysis | 1 | |

## Table 9-1. Summary of Analytical Procedures and Method

### Page 2 of 2

| Analysis | Sample Matrix | Field Samples | | | Reference Preparation Method | Reference Analytical Method | QC Analysis | QC Analysis Frequency | QC Analyses | Total Analyses |
|---|---|---|---|---|---|---|---|---|---|---|
| | | NCIN-1 Test 1 | NCIN-2 Test | Field QC | | | | | | |
| PCDD/PCDFs by SW846 Method 0023A (cont'd) | Method 0023A toluene reagent blank | -- | -- | 1 | NA | HRGC/HRMS for PCDD/PDDFs (SW-846 Method 8290) | PCDD/PCDF isotope dilution internal standard spike | Every analysis | 1 | 1 |
| | | | | | | | PCDD/PCDF recovery standard spike | Every analysis | 1 | |
| | Method 0023A spiked XAD-2 resin blank | -- | -- | 2 | Soxhlet extraction with MeCl2 (SW-846 Methods 0023A/3542, EPA Method 1668A) | HRGC/HRMS for PCDD/PDDFs (SW-846 Method 8290) | PCDD/PCDF pre-sampling surrogate spikes | Every XAD-2 resin tube before sampling | 2 | 2 |
| | | | | | | | PCDD/PCDF isotope dilution internal standard spike | Every analysis | 2 | |
| | | | | | | | PCDD/PCDF recovery standard spike | Every analysis | 2 | |
| | Method 0023A spiked XAD-2 resin (Audit Sample | -- | -- | 1 | Soxhlet extraction with MeCl2 (SW-846 Methods 0023A/3542) | HRGC/HRMS for PCDD/PDDFs (SW-846 Method 8290) | PCDD/PCDF pre-sampling surrogate spikes | Every XAD-2 resin | 1 | 1 |
| | | | | | | | PCDD/PCDF isotope dilution internal standard spike | Every analysis | 1 | |
| | | | | | | | PCDD/PCDF recovery standard spike | Every analysis | 1 | |
| | Analytical system QC | NA | | | Soxhlet extraction with MeCl2 (SW-846 Methods 0023A/3542) | HRGC/HRMS for PCDD/PDDFs (SW-846 Method 8290) | Method blank | 1 per analytical batch | 1 or more | 1 |
| | | | | | | | Blank spike | 2 per analytical batch | 2 or more | 2 |
| TOTAL | | | | | | | | | | 23 |

**Table 9-2. Stack Gas Dioxin/Furan Target Analytes**
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| Compound | CAS No. | Per Analysis EDL (pg) | Front Half Detection Limit (ng)[a,b] | Back Half Detection Limit (ng)[a,b] | Train Detection Limit (ng)[a,b] | Train Detection Limit (ng/m$^3$)[c] | 2,3,7,8-TCDD TEQ Factor | 2,3,7,8-TCDD TEQ Limit (ng/m$^3$)[c] |
|---|---|---|---|---|---|---|---|---|
| 2,3,7,8 - TCDD | 1746-01-6 | 1.8 | 0.0018 | 0.0018 | 0.0036 | 0.0012 | 1 | 0.0012 |
| Total TCDD | NA | 1.8 | 0.0018 | 0.0018 | 0.0036 | 0.0012 | NA | NA |
| 2,3,7,8 - TCDF (DB225) | 51207-31-9 | 1.9 | 0.0019 | 0.0019 | 0.0038 | 0.0013 | 0.1 | 0.00013 |
| Total TCDF | NA | 1.9 | 0.0019 | 0.0019 | 0.0038 | 0.0013 | NA | NA |
| 1,2,3,7,8 - PeCDD | 40321-76-4 | 1.8 | 0.0018 | 0.0018 | 0.0036 | 0.0012 | 0.5 | 0.00060 |
| Total PeCDD | NA | 1.8 | 0.0018 | 0.0018 | 0.0036 | 0.0012 | NA | NA |
| 1,2,3,7,8 - PeCDF | 57117-41-6 | 1.9 | 0.0019 | 0.0019 | 0.0038 | 0.0013 | 0.05 | 0.000063 |
| 2,3,4,7,8 - PeCDF | 57117-31-4 | 1.9 | 0.0019 | 0.0019 | 0.0038 | 0.0013 | 0.5 | 0.00063 |
| Total PeCDF | NA | 1.9 | 0.0019 | 0.0019 | 0.0038 | 0.0013 | NA | NA |
| 1,2,3,6,7,8 - HxCDD | 57653-85-7 | 2.2 | 0.0022 | 0.0022 | 0.0044 | 0.0015 | 0.1 | 0.00015 |
| 1,2,3,4,7,8 - HxCDD | 39227-28-6 | 1.7 | 0.0017 | 0.0017 | 0.0034 | 0.0011 | 0.1 | 0.00011 |
| 1,2,3,7,8,9 - HxCDD | 19408-74-3 | 2.0 | 0.0020 | 0.0020 | 0.0040 | 0.0013 | 0.1 | 0.00013 |
| Total HxCDD | NA | 1.9 | 0.0019 | 0.0019 | 0.0038 | 0.0013 | NA | NA |
| 1,2,3,6,7,8 - HxCDF | 57117-44-9 | 1.9 | 0.0019 | 0.0019 | 0.0038 | 0.0013 | 0.1 | 0.00013 |
| 1,2,3,4,7,8 - HxCDF | 70648-26-9 | 1.6 | 0.0016 | 0.0016 | 0.0032 | 0.0011 | 0.1 | 0.00011 |
| 1,2,3,7,8,9 - HxCDF | 72918-21-9 | 2.1 | 0.0021 | 0.0021 | 0.0042 | 0.0014 | 0.1 | 0.00014 |
| 2,3,4,6,7,8 - HxCDF | 60851-34-5 | 2.3 | 0.0023 | 0.0023 | 0.0046 | 0.0015 | 0.1 | 0.00015 |
| Total HxCDF | NA | 1.9 | 0.0019 | 0.0019 | 0.0038 | 0.0013 | NA | NA |
| 1,2,3,4,6,7,8 - HpCDD | 35822-39-4 | 3.1 | 0.0031 | 0.0031 | 0.0062 | 0.0021 | 0.01 | 0.000021 |
| Total HpCDD | NA | 3.1 | 0.0031 | 0.0031 | 0.0062 | 0.0021 | NA | NA |
| 1,2,3,4,6,7,8 - HpCDF | 67562-394 | 1.9 | 0.0019 | 0.0019 | 0.0038 | 0.0013 | 0.01 | 0.000013 |
| 1,2,3,4,7,8,9 - HpCDF | 55673-89-7 | 2.2 | 0.0022 | 0.0022 | 0.0044 | 0.0015 | 0.01 | 0.000015 |
| Total HpCDF | NA | 2.0 | 0.0020 | 0.0020 | 0.0040 | 0.0013 | NA | NA |
| Total OCDD | 3268-87-9 | 12 | 0.012 | 0.012 | 0.024 | 0.0080 | 0.001 | 0.0000080 |
| Total OCDF | 39001-02-0 | 4.2 | 0.0042 | 0.0042 | 0.0084 | 0.0028 | 0.001 | 0.0000028 |

**Table 9-2. Stack Gas Dioxin/Furan Target Analytes**

**Page 2 of 2**

| Compound | CAS No. | Per Analysis EDL (pg) | Front Half Detection Limit (ng)[a,b] | Back Half Detection Limit (ng)[a,b] | Train Detection Limit (ng)[a,b] | Train Detection Limit (ng/m$^3$)[c] | 2,3,7,8-TCDD TEQ Factor | 2,3,7,8-TCDD TEQ Limit (ng/m$^3$)[c] |
|---|---|---|---|---|---|---|---|---|
| **TOTALS** | | | | | | 0.022 | | 0.0036 |

Notes:

[a] Detection limits based on analysis of the separate front-half and back-half extracts. The detection limits values represent approximate, relative limits using isotope dilution and HRGS/HRMS analysis which can result in detection limit variations.

[b] Based on separate front and back extract analyses.

[c] Based on 3.0 cubic meters of stack gas sampled.

# 10.0 SPECIFIC INTERNAL QUALITY CONTROL CHECKS

## 10.1 DEFINITIONS

The various types of QA/QC checks that may be performed as part of the test, both for sampling and analysis, are defined below. One or more of these QA/QC checks are associated with each measurement system in order to assess the compliance of the data to the DQOs established in Section 5.0. Table 9-1 includes a summary of all the sample analyses and their associated internal quality control checks associated with this test program.

**Audit Sample** An audit sample is a field or alternate laboratory prepared blank spike submitted to the test laboratory to assess accuracy or potential sample degradation.

**Blank, Field** A field blank is a sampling train or sampling component that is set-up in the field but is not used for test sampling. The field blank is used to assess background contamination that may affect the representativeness of the field samples.

**Blank, Media** A sample of unused sampling media analyzed to ensure the media are uncontaminated. This type of sample may also be referred to as a "reagent blank" (see below).

**Blank, Method** A method blank is a sample of unused media that is prepared and analyzed in the test laboratory to assess background contamination that may exist in the laboratory, on glassware, or in the analytical system.

**Blank, Reagent** A sample of unused reagent(s) used to demonstrate the absence of contamination in the reagents.

**Blank, Spike** A blank spike is a laboratory prepared sample of blank media that is spiked with a known amount of target analyte(s) used to assess the accuracy of the analytical method.

**Blank, System** An aliquot of uncontaminated reagent used to clean out the analytical system after high level samples have been analyzed or before analysis begins.

**Blank, Trip** A trip blank is an unused sample component that is shipped to the field along with the sampling equipment/media and/or returned to the laboratory without having been exposed to field conditions. If contamination is encountered in the field blank(s), the trip blank is analyzed to assess whether or not the contamination originates in the field, is inherent in the equipment/media, or results from exposure during shipping and handling.

**Calibration Check** A standard solution from a source other than the calibration standards used to verify the integrity of an instrument's calibration.

**Calibration Standards** High purity compounds or mixtures of compounds used to adjust the response of an analytical instrument. The laboratory will use traceable standards and submit standard preparation logs as part of the deliverables package.

**Continuing Calibration Verification** A mid-point standard, from the same Calibration source as the initial calibration solution analyzed periodically to verify that calibration conditions have not drifted from the initial calibration.

**Initial Calibration** A series of analyses of solutions, that have known concentrations, used to establish the correspondence between the amount of an analyte present in the solution and the instrument's response across the expected analytical range of the samples. Initial calibrations also establish retention time windows for identification purposes in chromatographic methods.

**Internal Standard Recovery** Internal standards are non-target spikes added to samples for quantitation purposes. The percent recovery of the internal standards is checked to assess whether or not significant matrix interferences may affect the accuracy and precision of analytical results.

**Performance Evaluation (PE) Sample** See Audit Sample.

**Proficiency Test** A series of blank spikes analyzed in the test laboratory to demonstrate an analyst's ability to successfully perform the method with acceptable precision and accuracy.

**Replicate** One of a series of identical samples or splits of a single sample used to assess precision.

**Spike, Field** See Audit Sample.

**Spike, Matrix** Spike of the known or controlled amount of an actual target analyte to an actual sample matrix that is then analyzed for that analyte. The percent recovery of the spiked analyte provides a measure of the matrix bias.

**Surrogates** Non-target or isotopically labeled analytes spiked into field samples as a measure of method efficiency and accuracy.

## 10.2 SPECIFIC QUALITY CONTROL CHECKS AND ACCEPTANCE CRITERIA

A variety of QC checks are required both in the field and in the laboratory to ensure the collection of samples that accurately represent the field conditions under study, to assess compliance with the Data Quality Objectives (DQOs), and to assess biases in the measurement system.

### 10.2.1 Field Activities

In order to ensure the representativeness of samples collected during the test, and to ensure integrity of field measurements, a variety of QC checks and controls will be implemented throughout the sampling program. These checks and controls will include:

- Standard forms and/or standard field notebooks will be used to document field activities and for data collection. The data collection forms and field notebooks will be reviewed routinely by senior staff for accuracy, completeness, and internal consistency.
- The strict adherence to detailed operating procedures as documented in the various project controlling documents and related SOPs will be enforced by experienced senior technical staff.
- Project personnel will be selected based on appropriate levels of training and experience and will receive site-specific training prior to working on-site. The site-specific training will include health and safety requirements; security requirements; briefings on overall project goals, objectives, and schedules; and, specific technical training related to their assigned tasks.
- Routine calibration will be performed on measurement systems and sampling equipment including metering systems, thermocouples, barometers, rotameters, and pitot tubes. Guidance related to equipment calibration is provided in Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods and Quality Assurance/Quality Control Procedures for Hazardous Waste Incinerators, Appendix A. The detailed specifications, acceptance criteria, and corrective action requirements are presented in Section 8.0 of this QAPP. All calibrations will be documented and the documentation maintained in the project files.
- Leak checks will be performed according to method specifications before and after sampling.
- Field QC samples will be routinely submitted including audit (PE) samples, field blanks, media blanks, reagent blanks, trip blanks, and contingency samples. The frequency of submittal for these field QC samples and other field samples are provided in Tables 5-1 and 8-2.
- Field audits/surveillance will be performed periodically by the Hexion Test Project Manager to assess conformance to specifications. If nonconforming conditions are noted, the corrective action provisions of the QA plan will be invoked.

### 10.2.2 Laboratory Activities

Standard laboratory QA procedures, required of each laboratory, provide discussions related to QA/QC checks and controls within the laboratory. Specific data quality objectives, calibration requirements, acceptance criteria, and corrective action requirements for this test program are presented in Table 5-1 and Table 8-2 of this plan.

In addition to the requirements referenced above the laboratory will provide for quality control of sampling media and sample collection equipment. Sorbents used in the organic sampling trains will be prepared according to method specifications. Samples of the prepared media will be tested according to the intended method of use. The results of these tests will be retained in the laboratory's files for future reference.

# 11.0 DATA REDUCTION, DATA VALIDATION, AND DATA REPORTING

## 11.1 DATA REDUCTION

This section of the QAPP describes the approach that will be used to report, review, and reduce the field and laboratory data. The raw data include field samples and sampling documentation, sample tracking documentation, laboratory preparation documentation, and raw analytical data. The analytical results from the laboratory are assembled into complete analytical data packages. The analytical data packages include the analytical results and their defensible backup data. The analytical data are evaluated for compliance with the project DQOs. Data determined to meet the analytical requirements will be used to calculate the unit performance and emissions.

## 11.2 ORGANIC ANALYSES

Organic analyses will be conducted using gas chromatography (GC) techniques. Although the principles of operation and specific methods of calibration differ according to the analyte specific methods, the general data reduction scheme is the same for all of these tests. The individual methods should be consulted for details. A summary of the data reduction scheme is presented below.

Depending on the specific method, analytical instrumentation is calibrated using 3 to 5 points covering the expected analytical range. The gas chromatograph/flame ionization detector (GC/FID) or gas chromatograph/electron capture detector (GC/ECD) methods generally employ an external standard calibration technique while the GC/MS methods employ an internal standard technique. For GC/FID and GC/ECD methods, a calibration factor (CF) is calculated using the following formula:

$$CF = \frac{R}{M}$$

Where: $CF$ = Calibration Factor
$R$ = Response or Area of the GC Peak
$M$ = Mass Injected (in nanograms)

The calibration factors must agree to within method specified criteria for the percent relative standard deviation (%RSD). The formula for %RSD is given below.

$$\%\,\mathrm{RSD} = \frac{\sigma}{\mathrm{avg\,CF}} \times 100$$

Where: $\%RSD$ = Percent Relative Standard Deviation

$\sigma$ = Standard Deviation of the Calibration Factors

$avg\ CF$ = Average Calibration Factor

For gas chromatograph/mass spectral (GC/MS) calibrations, a response factor (RF) is used rather than the CF. The formula for the RF is:

$$RF = \frac{(A_s \times C_{is})}{(A_{is} \times C_s)}$$

Where: $RF$ = Response Factor

$A_s$ = Response for the Analyte

$A_{is}$ = Response for the Internal Standard

$C_s$ = Concentration of the Analyte

$C_{is}$ = Concentration of the Internal Standard

The RFs must also pass a test of the %RSD in order for the calibration to be considered valid.

When samples are analyzed, the area of the peak produced by a given analyte is compared to the CF or RF to arrive at an analytical result according to the following formula:

$$M_x = A_x \times CF$$

Where: $M_x$ = The Mass of Analyte in the Sample

$A_x$ = The Response of the Analyte in the Sample

$CF$ = The Calibration (or Response) Factor

Samples containing a concentration of an analyte exceeding the concentration the instrument is calibrated for, will have a dilution performed, if such a dilution is practical given the sample preparation method. In that case $M_x$ is multiplied by the dilution factor to arrive at the final result. If, under the circumstances of the method, a dilution is not possible, the analytical result must be considered estimated.

To arrive at a concentration in the gas sample the mass of any sub-samples must be added together and then compared to the volume of gas sampled according to the following formula:

$$C_x = \frac{(M_1 + M_2 + ...M_n)}{V}$$

Where: $C_x$ = The Concentration of the Analyte in the Gas Sample

$M_n$ = The Result (Mass) for Each Component in the Sampling Train

$V$ = The Volume of Gas Sampled

## 11.3 ANALYTICAL DATA PACKAGES

Analytical data packages will be organized in accordance with the laboratory standard operating procedures. The complete analytical data package is a stand-alone deliverable that includes the final analysis results, raw analytical instrument data, initial and continuing calibration data, parameter-specific quality control documentation, sample preparation documentation, and records of sample traceability. These data are sufficient for performing independent verification of the final analytical results. Every analytical data package includes the following:

- Cover Page—Identifies the laboratory-assigned lot number, project identification, laboratory project manager, and issue date.
- Table of Contents—Organization of the data package.
- Sample Summary—Cross reference to project sample identifications and laboratory sample identifications.
- Analytical/Preparation Methods Summary—Identifies the methods used to prepare and analyze the samples.
- Narrative— Summarizes the project-specific information and any pertinent information concerning data quality. The narrative documents sample delivery and condition upon receipt, and any analytical difficulties or anomalies encountered during sample preparation and analysis.
- QC Data Association Summary—Comparison of sample results to the project and laboratory DOQs and association of project samples to laboratory QC.
- Analytical Data Report—Summarized analytical data for all samples and associated quality assurance samples including as appropriate: data flags, duplicate analysis results, surrogate recovery results, method blank results, laboratory control sample results, and matrix spike (MS) results.
- Chain-of-custody (COC) documentation

Each analytical data package will include the identification and signature or initials of each analyst who handles the test samples. The Laboratory Analysis Coordinator will certify via signature the contents of each analytical data package.

### 11.3.1 Organic Analyses

The organic analytical data packages will include the following:

- Sample Results
- Raw Sample Data
  - Sample Data
- • Standards Data
  - Initial Calibration Summary(s) and Raw Data
  - Continuing Calibration Summary(s) and Raw Data
  - Initial Calibration(s) Tuning Raw Data (GC/MS only)
  - Continuing Calibration(s) Tuning Raw Data (GC/MS only)
- Raw QC Data
  - Method Blank Data
  - MS/MSD Data and Evaluation Reports
  - Laboratory Control Standard Data and Evaluation Reports
- Miscellaneous data
  - Sample Data Review Checklist
  - Calibration Data Review Checklists
  - Run Logs
  - Extraction sheets.
  - Sample Results
- Example Calculations

## 11.4 DATA VALIDATION

The results of all sample analysis and all QA/QC sample analysis (100% of the laboratory data) will be compared to the specifications given in Tables 5-1 and 8-2. The data validation criteria outlined in: USEPA Contract Laboratory Program National Functional Guidelines for Low Concentration Organic Data Review, (2001) and USEPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Review (2002) prepared by USEPA Data Review Work Group will be followed as applicable. Any sample data associated with a QC check that fails to meet the target criteria established in these tables will be flagged in the final report, and an assessment of the impact, if any, of missing the target data quality objective will be provided. Additional guidance will be found in the analytical methods and EPA/625/6-89/023, Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration.

Detailed procedures for the internal review of data in the laboratory are found in the laboratories QA Manuals and related standard operating procedures (SOPs).

## 11.5 DATA REPORTING

### 11.5.1 Project Reporting Format

The outlines for the final test reports are presented in Figures 11-1 and 11-2.

### 11.5.2 Detection Limit Definitions

Isotope dilution methods quantify non-detects using estimated detection limits (EDLs) as defined in SW-846 Method 8290. The EDL is defined as follows:

> EDL (from SW-846 Method 8290) -*"The sample specific estimated concentration of a given analyte required to produce a signal with a peak height of 2.5 times the background signal level."*

EDLs are sample-specific and analyte-specific, and will be provided on the analytical certificate for each sample and each analyte.

### 11.5.3 Detection Limits and Data Reduction

In accordance with 40 CFR 63.1208(b)(1)(iii), by operating the Method 0023A sampling train a minimum of 180 minute (3 hours) to sample a minimum of 2.5 dry standard cubic meters of stack gas during each sampling run, any dioxin/furan congener that is non-detect will be counted as zero in determining compliance. For the front-half and back-half separate analyses of the Method 0023A samples, if a fraction is non-detect for a specific congener, the specific congener's EDL will reported. In subsequent compliance calculations of the 2,3,7,8-TCDD toxicity equivalents, the individual congener concentrations for each fraction that are reported as non-detect will be counted as zero for that fraction.

### 11.5.4 Other Quality Control Data Reporting

Other quality control data that are included in the analytical reports and may be discussed or commented on in the final test reports are:

- Sample holding times
- Sample surrogate recovery results
- Duplicate results
- MS results
- Blank train results
- Reagent blank results (if analyzed)
- Trip and field blank results
- RPD and RSD evaluations of accuracy or precision.

These data will be evaluated against the target DQOs. Any data that fall outside of the DQO limits will be flagged, footnoted, and assessed relative to the performance and emissions testing objectives. The data from the field blanks and trip blanks will be used to assess possible contamination from field handling and

transport of the samples to the laboratory. The reagent blank and blank train results will be used to assess contamination resulting from reagents used, sample handling, and sample recovery procedures. Any blank results that may have impacted performance and emissions testing results or evaluations will be flagged, footnoted, and discussed in the test reports.

### 11.5.5 Final Case Files

At a minimum, the following documents will be retained upon the completion of the project in the final case file, which must be maintained at the Hexion facility for a period at least three years:

- All legal documents and orders,
- All field documents including those used for preliminary field activities,
- Copies of all analytical data,
- Copies of the final report and background documents, and
- All correspondence relating to the project as well as corrective action requests.

**Figure 11-1. Example Confirmatory PCDD/PCDF Test Report Outline**

CERTIFICATION FORM *(LAC 33:V.513.)*
TABLE OF CONTENTS
LIST OF FIGURES
LIST OF TABLES
LIST OF APPENDICES
**1.0** SUMMARY OF TEST RESULTS
- **1.1** PCDD/PCDF Confirmatory Test Results
- **1.2** Deviations from the Approved Confirmatory Test Plan and Their Impacts
- **1.3** Actual vs. Planned Operations
- **1.4** Data Quality

**2.0** INTRODUCTION/PROCESS DESCRIPTION
- **2.1** Brief Unit Description
- **2.2** Test Objectives Overview
- **2.3** Test Responsible Parties
- **2.4** Test Chronology
- **2.5** HWC MACT Process Monitors

**3.0** OPERATING DATA SUMMARY
**4.0** EMISSIONS RESULTS
- **4.1** Dioxin/Furan Emission Results
- **4.2** CEMS Monitoring Results for Carbon Monoxide, Total Hydrocarbons, and Oxygen

**5.0** QUALITY ASSURANCE/QUALITY CONTROL DOCUMENTATION
- **5.1** Quality Assurance/Quality Control Objective Results
- **5.2** Performance Evaluation (Audit) Results
- **5.3** Sample Handling, Traceability, and Holding Time Results
- **5.4** Calibration Results
  - **5.4.1** Process Monitoring Equipment
  - **5.4.2** Stack Sampling Equipment
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APPENDICES
- APPENDIX A PROCESS OPERATING DATA
- APPENDIX B STACK SAMPLING REPORT
- APPENDIX C PROCESS INSTRUMENT CALIBRATIONS
- APPENDIX D SAMPLE CALCULATIONS
- APPENDIX E FIELD NOTES AND LIST OF SAMPLES
- APPENDIX F TEST TEAM RESUMES
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- APPENDIX J LELAP APPROVALS
- APPENDIX K AUDIT SAMPLE RESULTS

# 12.0 ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES

## 12.1 SAMPLING EQUIPMENT

All equipment used in emission testing measuring systems must be maintained in good operating order. To achieve this objective, a routine preventive maintenance program is necessary. Procedures used in this program follow those outlined in Maintenance Calibration and Operation of Isokinetic Source Sampling Equipment, Publication No. APTD-05-76 and Volume III of the Quality Assurance Handbook for Air Pollution Measurement Systems.

The potential impact of equipment malfunction on data completeness is minimized through two complementary approaches. First, an equipment maintenance program is part of routine operations. The maintenance program's strengths include:

- Trained technicians experienced in the details of equipment maintenance and fabrication,
- Adequate spare parts inventory, and
- The availability of tools and specialized equipment.

The second approach is based upon equipment redundancy. Backup equipment, spare parts and tools are included on the materials transported to the field for each sampling task. This approach allows the sampling team to respond to equipment breakage or malfunction in a timely fashion, minimizing the quantity of lost data.

For field equipment, preventive maintenance schedules are based on the results of routine inspections and on accumulated experience. At a minimum, equipment will be inspected prior to the beginning of and at the conclusion of each test. A record of each inspection (Figure 12-1) will be kept as part of the final case file. Maintenance schedules for continuous emissions monitors follow manufacturer's recommendations.

Each item of field test equipment is assigned a unique, permanent identification number. An effective preventive maintenance program is necessary to ensure data quality. Each item of equipment returning from the field is inspected before it is returned to storage. During the course of these inspections, items are cleaned, repaired, reconditioned and recalibrated where necessary. Each item of equipment transported to the field for this test program is inspected again before being packed to detect equipment problems that may originate during periods of storage. This minimizes lost time on the job site due to equipment failure. Occasional equipment failure in the field is unavoidable despite the most rigorous

inspection and maintenance procedures. For this reason, adequate spare parts are kept in a central location so the sampling contractor can quickly respond to the job site with replacement equipment for all critical sampling train components.

## 12.2 LABORATORY INSTRUMENTS

The laboratories perform regular maintenance on all analytical instruments. An inventory of replacement parts is kept to prevent downtime. Manufacturers' service representatives are also contracted, as required, for major instrument repairs.

Preventive and routine maintenance is covered in each of the laboratories' QA Manuals and SOPs or in accordance with manufacturer's recommendations (i.e., instrument manuals). Daily maintenance (such as replacement of injector septa, etc.) is covered in instrument SOPs. Inoperative equipment is tagged as non-usable until repairs are performed. Logbooks are maintained for each instrument to record usage, maintenance, and repairs.

## 12.3 PROCESS INSTRUMENTS

On-site personnel perform regular maintenance on all process instrumentation. Routine and preventive maintenance programs are used. Where appropriate, manufacturers' recommendations for maintenance of process instruments are followed. Operators conduct daily reviews of process instrumentation by noting suspicious or inconsistent readings. Maintenance logs are used to record the frequency and type of repairs necessary for process instruments. Process instruments used to demonstrate compliance with operating limits will be calibrated prior to the test. Records of these calibrations will be included in the final test report.

## Equipment Inspection Record

**Hexion Specialty Chemicals, Norco, LA**

Date/Time of inspection: _____

Equipment Inspected:

1) _____

Condition: Good See Problems Section Below

2) _____

Condition: Good See Problems Section Below

3) _____

Condition: Good See Problems Section Below

4) _____

Condition: Good See Problems Section Below

5) _____

Condition: Good See Problems Section Below

Problems Noted:

_____

_____

_____

_____

Action Taken

_____

_____

_____

Inspector's Signature_____

**Figure 12-1. Example Equipment Inspection Record Form**

## 13.0 ASSESSMENT PROCEDURES FOR ACCURACY, PRECISION, & COMPLETENESS

The QA activities implemented in this study will provide a basis for assessing the accuracy and precision of the analytical measurements. Section 5.0 discusses the QA activities that will generate the accuracy and precision data for each sample type. The generalized forms of the equations that will be used to calculate accuracy and precision are presented below.

### 13.1 ACCURACY

When a reference standard material is used in the analysis, percent Accuracy (A) will be calculated as follows:

$$A = \frac{\text{Found concentration}}{\text{True concentration}} \times 100$$

Percent analyte Recovery (R) will be calculated as follows:

$$R = \frac{X - N}{S} \times 100$$

Where X is the experimentally determined value, N is the amount of native material in the sample, and S is the amount of spiked material of the species being measured. Recoveries are used to determine accuracy when standards are not available, or are not appropriate for a given matrix.

### 13.2 PRECISION

When less than three analyses of the same parameter are available, precision will be calculated as a Relative Percent Difference (RPD) from the average of replicate measurements according to:

$$\text{RPD} = \frac{(X_1 - X_2)}{\text{Average } X} \times 100$$

Where $X_1$ and $X_2$ are the highest and lowest results of replicate measurements.

Where three or more analyses of the same parameter are available, the precision will be determined as the Relative Standard Deviation (RSD) according to:

$$\text{RSD} = \frac{\text{Standard deviation}}{\text{Average X}} \times 100$$

**13.3 COMPLETENESS**

Completeness of data generated from a test program is usually calculated as follows:

$$\%\ \text{Completeness} = \frac{\text{Valid data}}{\text{Expected data}} \times 100$$

Data completeness is defined in Section 5.0 of this QAPP as the percentage of valid data collected from the total number of valid tests conducted. Three valid test runs, at each test condition, are required for the test to be completed. If an individual sample from a test run is lost or broken, the data for that individual analytical parameter may not be 100% complete. This, however, may not invalidate the test run. The completeness objective for this test program is to generate sufficient data for the regulatory agencies to judge the performance of the system.

# 14.0 AUDIT PROCEDURES, CORRECTIVE ACTION, AND QA REPORTING

## 14.1 PERFORMANCE AND SYSTEM AUDITS

This section presents information related to the procedures used to assess conformance of the project staff to the specifications contained in the relevant project controlling documents. Further, auditing may be employed to assess the ability of subcontractors to successfully perform the work.

### 14.1.1 Field Audits

The Hexion Test Project Manager will continuously evaluate the operations at the site to ensure that work is being performed in accordance with the various project controlling documents and associated standard operating procedures. The field audit will cover, but is not necessarily be limited to, such areas as:

- Conformance to SOPs
- Completeness and accuracy of documentation
- Chain of custody procedures
- Compliance with Health and Safety requirements.

### 14.1.2 Performance Evaluations

The regulatory agency may submit Performance Evaluation (PE) samples (referred to elsewhere as "audit samples") to the laboratory. PE samples provided will be submitted for analysis to demonstrate analytical performance on an as required basis. Performance audit samples for PCDD/PCDF analyses will be requested from the EPA Regions 6, Ms. Esther Coleman [(214) 665-8517 or coleman.esther@epa.gov]

### 14.1.3 Laboratory Audits

Hexion or its appointed representative may choose to audit the laboratories at any time during the course of the project on an as-required basis to assess the laboratory's ability to successfully perform the work and to ensure mutual agreement between Hexion and the laboratory with regard to the scope of work, QA/QC requirements, and deliverable requirements. Reasonable notice will be provided prior to any on-site inspection of the laboratory.

## 14.2 CORRECTIVE ACTION

The following procedures have been established to ensure that nonconforming conditions, such as malfunctions, deficiencies, deviations and errors are promptly investigated, documented, evaluated and corrected. Every person employed in the test is expected to function as a QC inspector to ensure the quality of the final product. Quality, as it relates to this project, is defined as "performing the work according to the agreed upon specifications contained in the CPT Plan and relevant SOPs or causing the

specification to be changed *in a controlled manner.*" Each individual is encouraged to identify any condition adverse to the successful completion of the work or any modification to the specifications that might result in a better end product. These improvements might be framed in terms of higher quality, greater safety, greater efficiency, and/or lower cost.

### 14.2.1 Field

When a nonconforming condition or an opportunity for improvement is noted at the site or contractor location, the corrective action provisions of this plan will be invoked to identify the condition and recommend corrective action. Condition identification, cause, reference documents and the corrective action planned to be taken will be documented and reported at a minimum to the employee's immediate supervisor.

A Corrective Action Request (CAR), as shown in Figure 14-1, should be used to identify the adverse condition or opportunity for improvement, reference document(s) and recommended corrective action(s). The CAR is directed to the Hexion Test Project Manager. The Hexion Test Project Manager affixes his signature and the date to the corrective action block that states the cause of the condition(s) and corrective action(s) to be taken. The Hexion Test Project Manager is responsible for first notifying the regulatory agency representative of any problems or deviations from the QAPP, or the test plans identified in the CAR. The Hexion Test Project Manager performs follow-up where appropriate. CARs are transmitted to the project file for future reference, and are incorporated into the test reports.

### 14.2.2 Laboratory

The laboratories' QA Manuals and the related SOPs, contain detailed discussions of corrective actions to be taken if established criteria fail during laboratory analysis. The laboratory has the responsibility to immediately notify the Hexion Test Project Manager and the Stack Sampling Coordinator when any analytical QC nonconformance occurs, so a mutually acceptable course of action can be pursued.

## Corrective Action Request

Number:
Date:
File Name:
Client: Hexion Specialty Chemicals, Inc., Norco, LA

**REQUEST**

To:

You are hereby requested to take corrective actions indicated below and as otherwise determined by you (A) to resolve the noted condition and (B) to prevent it from recurring. Your written response is to be returned to the project Quality Assurance Officer or other responsible manager by.

**Condition:**

**Reference Documents:**

**Recommended Corrective Actions:**

| Originator | Date | Approval | Date | Approval | Date |
|---|---|---|---|---|---|

**RESPONSE**

**Cause of Condition:**

**Resolution:**

**Prevention:**

**Affected Documents:**

| | |
|---|---|
| Corrective Action Initiator | Date |
| Test Project Manager | Date |
| Test Project Manager Follow-up | Date |

**Figure 14-1. Example Corrective Action Request Form**

# ATTACHMENT A

# RESUMES OF KEY INDIVIDUALS

**RESUMES OF KEY INDIVIDUALS WILL BE PROVIDED**

**WHEN THE TEST TEAM IS SELECTED AND CONTRACTED**

# ATTACHMENT B
# LELAP APPROVALS

**LELAP APPROVALS OF THE SELECTED SAMPLING AND ANALYTICAL CONTRACTORS WILL BE PROVIDED WHEN THE TEST TEAM IS SELECTED**

# CONTINUOUS MONITORING SYSTEM PERFORMANCE EVALUATION TEST PLAN FOR NCIN-1 AND NCIN-2 INCINERATION SYSTEMS HEXION SPECIALTY CHEMICALS, INC. EPA I.D. NO. LAD 980 622 104

**PREPARED FOR:**

HEXION™
Specialty Chemicals

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**OCTOBER 2008**
**Revision: 0**
**Focus Project No. 060804**

**PREPARED BY:**

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## APPENDIX A: EXAMPLE CALIBRATION DATA SHEETS

## ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| AWFCO | automatic waste feed cutoff |
| CD | calibration drift |
| CE | calibration error |
| CEM | continuous emissions monitoring |
| CEM PET | CEM Performance Evaluation Test |
| CEMS | CEM system |
| CPT | Comprehensive Performance Test |
| CMS | continuous monitoring system |
| CMS PEP | CMS Performance Evaluation Plan |
| CMS PET | CMS Performance Evaluation Test |
| CMS PETP | CMS Performance Evaluation Test Plan |
| CO | carbon monoxide |
| DCS | distributive control system |
| DOC | Documentation of Compliance |
| EPA | U.S. Environmental Protection Agency |
| HC or THC | total hydrocarbons |
| HWC MACT | Hazardous Waste Combustor Maximum Achievable Control Technology |
| LDEQ | Louisiana Department of Environmental Quality |
| MACT | Maximum Achievable Control Technology |
| NESHAP | National Emission Standards for Hazardous Air Pollutants |
| NCIN-1 | Norco Organic Chloride Incinerator Number One |
| NCIN-2 | Norco Organic Chloride Incinerator Number Two |
| NDIR | non-dispersive infrared |
| NOC | Notification of Compliance |
| $O_2$ | oxygen |
| QA | quality assurance |
| RA | relative accuracy |
| RCRA | Resource Conservation and Recovery Act |
| RM | reference method |
| RT | response time |
| VAX | VAX computer system |

# 1.0 INTRODUCTION

Hexion Specialty Chemicals, Inc. (Hexion) operates two hazardous waste incineration systems, Norco Organic Chloride Incinerators Numbers One and Two (NCIN-1 and NCIN-2), at the Norco, Louisiana chemical manufacturing facility. Each incineration system includes a continuous monitoring system (CMS) for demonstrating continuous compliance with applicable emission standards and operating parameters. Each CMS includes "parameter CMS" such as flowmeters, pressure transmitters, thermocouples, pH meters, etc., that measure parameter data from the operations of the incineration system, and a continuous emission monitoring system (CEMS) that monitors stack gas concentrations of carbon monoxide (CO) and oxygen ($O_2$).

On September 30, 1999, U.S. Environmental Protection Agency (EPA) promulgated the Final National Emission Standards for Hazardous Air Pollutants (NESHAP) for Hazardous Waste Combustors, otherwise known as the Hazardous Waste Combustor Maximum Achievable Control Technology (HWC MACT) rule. This rule applies to incineration systems that burn hazardous waste. The HWC MACT is promulgated at 40 CFR Part 63 Subpart EEE.

The HWC MACT places considerable emphasis on continuous monitoring to demonstrate compliance with applicable emission standards and operating parameter limits. As such, facilities are required to conduct performance evaluations on the CMS used to demonstrate compliance with the HWC MACT requirements. As part of the miniburn testing and Comprehensive Performance Test (CPT) for NCIN-1 and NCIN-2, Hexion will conduct a CMS Performance Evaluation Test (CMS PET) to verify the CMS are calibrated and collecting valid data.

This CMS Performance Evaluation Test Plan (CMS PETP) is submitted as an Appendix to the CPT Plan. It includes the detailed procedures and frequencies for calibration and maintenance of the parameter CMS (flow, temperature, pressure, etc.), and CO and $O_2$ CEMS. This plan will be in place as part of the modified incineration systems' operating record when compliance with the HWC MACT regulations is certified.

## 2.0 PERFORMANCE EVALUATION OBJECTIVES

The purpose of the CMS PET is to assess the accuracy of the instruments used to continuously demonstrate compliance with the HWC MACT requirements. This assessment includes ensuring the instruments are properly integrated with the incinerator automatic waste feed cutoff (AWFCO) system. To verify the accuracy of an instrument, Hexion will demonstrate that the instrument meets either the performance specification promulgated by EPA, or the manufacturer's recommended specification if an appropriate performance specification has not been promulgated. Hexion will also perform an AWFCO system test to verify that an alarm will be triggered and the hazardous waste feed will be shut off if the control system records an operating parameter outside the AWFCO setpoint. The performance evaluation program summary is discussed in more detail in Section 5.0. Specific performance criteria are also discussed in Section 5.0 of this plan.

## 3.0 PERFORMANCE EVALUATION TEST PLAN OBJECTIVES

Pursuant to Section 63.1207(e) of the HWC MACT, Hexion must develop and submit a site-specific CMS PETP for approval by the Administrator. The purpose of the CMS PETP is to provide a synopsis of the methods and procedures that will be used to verify the monitoring devices included in the CMS are calibrated and collecting valid data during unit operation. The objectives of this CMS PETP are to:

- Describe the CMS
- Outline the methods and procedures that will be used to calibrate the CMS
- Specify instrument performance criteria
- Describe the CMS integration with the AWFCO system
- Outline the AWFCO functional testing
- Present a schedule for the program procedures, tests, and audits
- List the data quality objectives
- Present the internal and external quality assurance (QA) program

Specific procedures referenced and summarized by this test plan for NCIN-1 and NCIN-2 will be included in the CMS Performance Evaluation Plan (CMS PEP) that will be developed pursuant to Section 63.8(d) of the MACT General Provisions and included in the operating record.

# 4.0 CONTINUOUS MONITORING SYSTEM (CMS) DESCRIPTION

The CMS for Hexion's NCIN-1 and NCIN-2 will consist of parameter CMS and CEMS. Parameter CMS are instruments that are electronically connected to the distributive control system (DCS). The data collected by the DCS are stored and managed by the connected VAX computer system (VAX). The VAX performs the data processing necessary to demonstrate compliance with system operational limits. The CEMS includes monitors that measure CO and $O_2$ concentrations in the stack to demonstrate compliance with the CO emission limit. Figures 4-1 and 4-2 show the general monitoring locations for temperature, pressure, flow and other measurement instruments related to the each incinerator's AWFCO system. Further description is provided below.

## 4.1 PARAMETER CMS DESCRIPTION

Parameter CMS consist of a combination of instruments that continuously monitor and record parameter data from the operations of the incineration systems. A list of the continuously monitored HWC MACT operating parameters applicable to NCIN-1 and NCIN-2 is presented in Table 4-1. The parameters are grouped consistent with the CPT Plan:

- Group 1 parameter limits are established from test operating data, and are used to ensure that incineration system operating conditions are not significantly less rigorous than those demonstrated during the test. Group 1 parameters are continuously monitored and recorded, and are interlocked with the automatic waste feed cutoff system.

- Group 2 parameter limits are regulatory specified limits, and are not based on the test operating conditions, e.g., the maximum stack CO or hydrocarbon (HC) concentration. Group 2 parameters are continuously monitored and recorded, and are interlocked with the automatic waste feed cutoff system.

- Group 3 parameter limits are based on manufacturer's recommendations, operational safety, and good operating practice considerations rather than on the test operating conditions. Group 3 parameter limits may be regulatory specified limits. Group 3 parameters may be continuously monitored and recorded, and may be interlocked with the automatic waste feed cutoff system.

### 4.1.1 CMS Instruments

Installed flowmeters, pressure transducers, thermocouples, and pH meters are used to collect process information on the incineration systems' key regulatory parameters. These key process monitoring instruments are listed in Tables 4-2 and 4-3 along with the instrument ranges for NCIN-1 and NCIN-2, respectively.

### 4.1.2 Data Acquisition and Recording

Each monitoring instrument produces a continuous electronic signal proportional to the instrument range. The signals are output to the incinerator DCS. The connected VAX uses the DCS collected data and an internal program to conduct required data manipulations and calculations. The VAX comuter compares

instantaneous or computed rolling average values to the respective AWFCO setpoints. The operating parameter data are stored in the VAX's data storage system.

The VAX evaluates each CMS measurement at least once every 15 seconds, and each minute computes and records the one-minute average value for each parameter from the four most recent 15-second readings. This meets the definition of a continuous monitor as defined by Section 63.1201(a) of the HWC MACT. To calculate an hourly rolling average, the VAX computes the arithmetic mean of the 60 most recent one-minute average values. As each new one-minute average value is recorded, the least recent of the previous 60 values is discarded from the data register, the new one-minute value is added, and an hourly rolling average is calculated and recorded. For a 12-hour rolling average, the arithmetic mean of 720 one-minute average values is calculated in the same way.

The VAX does not incorporate readings (data) generated during calibration periods into the instantaneous readings or the rolling average calculations. Once valid CMS data are available again, the VAX will resume rolling average calculations using the previously valid data values.

## 4.2 CONTINUOUS EMISSIONS MONITORING SYSTEM (CEMS)

The NCIN-1 and NCIN-2 stack gases are continuously monitored for CO and $O_2$ to indicate proper operation of the combustion process using the following procedures:

- Stack gas CO by non-dispersive infrared (NDIR) analyzer operated according to the protocols of 40 CFR 60, Appendix B, Specification 4B.
- Stack gas $O_2$ by paramagnetic analyzer operated according to the protocols of 40 CFR 60, Appendix B, Specification 4B.

CEMS for NCIN-1 AND NCIN-2 are extractive systems designed to continuously measure CO and $O_2$ levels in the exhaust gases. The ranges of the CEMS analyzers are provided in Tables 4-2 and 4-3 for NCIN-1 and NCIN-2, respectively. Each extractive sample conditioning system includes a sample probe and filter, a heated sample transport line, a sample conditioner, and a sample gas pump. Samples of the stack gas are continuously drawn through the sample probe located in the stack and in direct contact with the stack gas. The heated sample line transports the gas sample from the sample probe to the sample gas conditioner at a temperature above the sample gas dew point. The sample gas pump draws the continuous sample through the sample probe, the heated sample line and sample gas conditioner to the analyzers. In the sample gas conditioner, moisture is removed from the gas sample by first chilling the gas well below its dew point and condensing the moisture. The condensate is separated from the sample gas and the dry gas sample then enters the analyzers.

**Table 4-1. NCIN-1 and NCIN-2 HWC MACT AWFCO Parameters**

| Operational Parameter | Units | Averaging Period |
|---|---|---|
| **Group 1 Parameters** | | |
| Maximum liquid waste feed rate | lb/hr | Hourly Rolling Average |
| Minimum combustion temperature | °F | Hourly Rolling Average |
| Minimum caustic scrubber recycle flow | gpm | Hourly Rolling Average |
| Minimum caustic scrubber recycle pH | pH | Hourly Rolling Average |
| Maximum caustic scrubber recycle conductivity | µS/cm | 12-Hour Rolling Average |
| **Group 1 Parameters (continued)** | | |
| Minimum CATOX inlet gas temperature | °F | Hourly Rolling Average |
| Maximum stack gas flow | mscfm | Hourly Rolling Average |
| **Group 2 Parameters** | | |
| Maximum combustion chamber pressure | inwc | None; 1-second delay |
| Maximum stack gas CO concentration | ppmv, dry @ 7% $O_2$ | Hourly Rolling Average |
| **Group 3 Parameters** | | |
| Minimum caustic scrubber recycle pressure | psig | Hourly Rolling Average |
| Maximum CATOX inlet gas temperature | °F | Hourly Rolling Average |

**Table 4-2. NCIN-1 Key Regulatory CMS Instrumentation**

| Parameter | Units | Location [a] | Inst. ID | Instrument Type | Range |
|---|---|---|---|---|---|
| ACHE Feed Rate | lb/hr | F1 | FC-0551A | Mass Flow Meter | 0-10000 |
| TCP Feed Rate | lb/hr | F2 | FC-5533 | Mass Flow Meter | 0-10000 |
| HPRU Feed Rate | lb/hr | F3 | FC-5058 | Mass Flow Meter | 0-7500 |
| Combined Liquid Feed Rate | lb/hr | F1+F2+F3 | NA | Flow Totalizer | NA |
| Process Vent Gas Flow [b] | scfm | F4 | FI-5057 | Differential Pressure | 0-600 |
| Atomizing Steam Pressure | psig | P1 | Local PI | Pressure Gage | 0-100 |
| Firebox Pressure | inwc | P2 | PI-0565A | Pressure Transducer/ Transmitter | -1 to +1 |
| Firebox Temperature | °F | T1 | TC-0551 | Thermocouple | 0-2400 |
| Caustic Scrubber Recycle Flow | gpm | F5 | FI-0599 | Magnetic Flow meter | 0-650 |
| Caustic Scrubber Recycle pH | pH | A1 | AC-0524, AI-0525 | pH probe and Transmitter | 0-14 |
| Caustic Scrubber Recycle Conductivity | µS/cm | A2 | AI-5592 | Conductivity Cell | 0-25000 |
| Caustic Scrubber Recycle Pressure Bottom Distribution | psig | P3 | PI-5631 | Pressure Transducer/ | 0-50 |
| Caustic Scrubber Recycle Pressure Top Distribution | psig | P4 | PI-5636 | Pressure Transducer/ | 0-50 |
| CATOX Inlet Gas Temperature | °F | T2 | TT-5553 | Thermocouple | 0-1000 |
| Stack Gas Flow | mscfm | F6 | FF-5526 | Ultrasonic | 0-20 |
| Stack Gas CO Concentration | ppmv, dry | A3 | AI-0552 | Non-dispersive Infra-Red (NDIR) | 0-3000 |
| Stack Gas $O_2$ Concentration [c] | Vol. %, dry | A4 | AI-0551 | Paramagnetic Detector (PMD) | 0-25 |

Notes:
[a] Refer to Figure 4-1.
[b] Not interlocked with the AWFCO system; process vent flow rate information is necessary for determining the total feed rates of chloride [40 CFR 63.1209(c)(4)].
[c] Not interlocked with the AWFCO system; required for continuous correction of stack gas carbon monoxide concentration only.

NA = Not applicable

**Table 4-3. NCIN-2 Key Regulatory CMS Instrumentation**

| Parameter | Units | Location [a] | Inst. ID | Instrument Type | Range |
|---|---|---|---|---|---|
| ACHE Feed Rate | lb/hr | F1 | FC-5001 | Mass Flow Meter | 0-10000 |
| TCP Feed Rate | lb/hr | F2 | FC-5081 | Mass Flow Meter | 0-10000 |
| Combined Liquid Feed Rate | lb/hr | F1+F2 | NA | Flow Totalizer | NA |
| Process Vent Gas Flow [b] | scfm | F3 | FI-5029 | Differential Pressure | 0-600 |
| Atomizing Steam Pressure | psig | P1 | Local PI PC-5025 | Pressure Gage | 0-100 |
| Firebox Pressure | inwc | P2 | PI-5042A | Pressure Transducer/ Transmitter | -1 to +1 |
| Firebox Temperature | °F | T1 | TC-5001 | Thermocouple | 0-2400 |
| Caustic Scrubber Recycle Flow | gpm | F4 | FI-5069A | Magnetic Flow Meter | 0-650 |
| Caustic Scrubber Recycle Flow | gpm | F5 | FI-5070A | Magnetic Flow Meter | 0-650 |
| Recycle pH | pH | A1 | AC-5008, AI-5010 | pH probe and Transmitter | 0-14 |
| Recycle Conductivity | µS/cm | A2 | AI-5111 | Conductivity Cell | 0-25000 |
| Caustic Scrubber Recycle Pressure | psig | P3, P4 | PI-5122 PI-5123 | Pressure Transducers/ Transmitters | 0-150 |
| CATOX Inlet Temperature | °F | T2 | TT-5100 | Thermocouple | 0-1000 |
| Stack gas flow | mscfm | F6 | FF-5068 | Ultrasonic | 0-20 |
| Stack CO Concentration | ppmv, dry | A3 | AI-5009 | Non-dispersive Infra-Red (NDIR) | 0-3000 |
| Stack $O_2$ Concentration [c] | Vol. %, dry | A4 | AI-5002 | Paramagnetic Detector (PMD) | 0-25 |

Notes:

[a] Refer to Figure 4-2.

[b] Not interlocked with the AWFCO system; process vent flow rate information is necessary for determining the total feed rates of chloride [40 CFR 63.1209(c)(4)].

[c] Not interlocked with the AWFCO system; required for continuous correction of stack gas carbon monoxide concentration only.

NA = Not applicable

**Figure 4-1. NCIN-1 Monitoring Locations**

**Figure 4-2. NCIN-2 Monitoring Locations**

# 5.0 PERFORMANCE EVALUATION PROGRAM SUMMARY

The purpose of the CMS PET is to verify the CMS are calibrated and collecting valid data during the Miniburn/CPT. To calibrate the system instruments, Hexion will follow procedures defined in performance specifications promulgated by EPA, or manufacturer's recommendations for those devices that do not have promulgated performance specifications. The CMS PET of the CMS components will consist of three parts:

- Instrument calibration verification
- Functional testing of the AWFCO system and associated alarms, and
- Audit of CMS data reduction and recording functions.

A brief description of each is provided below.

## 5.1 INSTRUMENT CALIBRATION VERIFICATION

### 5.1.1 Parameter CMS

The parameter CMS will be calibrated as part of the CMS PET. Summaries of the calibration procedures are shown in Table 5-1.

#### 5.1.1.1 Instrument Calibration

The instrument calibration will be completed within 60 days prior to beginning the CPT. As EPA has not promulgated any specific performance specifications for parameter CMS, Hexion will use manufacturer's procedures and working knowledge of specific instruments to conduct the calibrations.

#### 5.1.1.2 Instrument Location

Pursuant to Section 63.8(c)(2) of the MACT General Provisions, all CMS shall be installed such that representative measurements of emissions or process parameters from the affected source are obtained. Hexion has installed all CMS so that they measure representative process parameters or process emissions. Instrument locations are shown in Figures 4-1 and 4-2 for NCIN-1 and NICN-2, respectively.

## 5.2 CEMS

The CEMS PET will follow the requirements in Performance Specification 4B for CO and $O_2$ CEMS found in 40 CFR Part 60, Appendix B. The CEMS PET will consist of the following four tests:

- Calibration Drift (CD) Test - Measures the difference in the CEM output readings compared to the actual calibration gas values after a 24-hour period of operation. This test demonstrates the stability of the CEMS over time.

- Calibration Error (CE) Test - Measures the difference between the concentration indicated by the CEM and the known concentrations of calibration gases. A CE test will document the accuracy and linearity of the monitoring equipment over the entire measurement range.

- Relative Accuracy (RA) Test - A measure of agreement between a measured value and an accepted or true value. The RA test is conducted by comparing the CEMS response to a value determined by using a standard reference method (RM) on a separate sampling and analytical system. The RA test is used to validate the calibration technique and verify the ability of the CEMS to provide representative and accurate measurements.

- Response Time (RT) Test - Measures the time interval between the start of a step change in the system input (e.g. change of calibration gas) and the time the data recorder displays 95% of the final value.

The CD, CE and RT tests are conducted separately on the CO and $O_2$ monitors. The RA test is conducted on a combined CO and $O_2$ reading (i.e., CO corrected to 7% $O_2$) such that separate RA tests are not required for these monitors. A summary of the performance criteria that must be demonstrated during this CEM PET is provided in Table 5-2. A brief description of each test is provided below.

### 5.2.1 Calibration Drift (CD) Test

The CD test must be conducted separately for each monitor while the facility is operating under normal conditions. The CD test will be conducted by determining the monitor response to the injection of zero-level and high-level calibration gas values. The zero-level and high-level calibration gas values to be used for CEMS are presented in Table 5-3. The CD readings are repeated every 24 hours for seven consecutive days. During this seven-day period, no unscheduled CEMS maintenance or repairs will take place. If the combustion unit is taken out of service during the test period, the onset and duration of the downtime will be recorded and the CD test will be continued when the unit resumes operation. Once the 24-hour drift reading has been recorded, the analyzer may be adjusted, if necessary.

#### 5.2.1.1 Carbon Monoxide Monitor

Data sheets will be used to record test data and calibration drift results for the CO monitor operating on the low span and the high span range. The CEMS test operator records the CO gas standard concentrations from the gas cylinders on the data sheet, and confirms that the concentrations are within the ranges shown in Table 5-3 for conducting the calibration drift test.

The zero-level and high span-level calibration gases are injected at the sample probe and the monitor responses to the gases are recorded on the data sheet. (Note that the monitor response recorded is not corrected to 7% oxygen.) The difference between the monitor response and the calibration gas value is the calibration drift test result, which is recorded on the data sheet. The calibration drift as a percent of span is calculated and recorded on the data sheet. The CO monitor calibration must not drift more than

an average of 3% from the reference standard (calibration gas value) for six out of seven consecutive test days.

#### 5.2.1.2 Oxygen Monitor

A data sheet is prepared for recording test data and calibration drift results for the $O_2$ monitor. The CEMS test operator obtains the $O_2$ gas standard concentrations (zero-level and span-level) from the gas cylinders, records the concentrations on the data sheet, and confirms that the concentrations are within the ranges shown in Table 5-3 for conducting the calibration drift test.

The zero-level and high span-level calibration gases are injected at the sample probe and the monitor responses to the gases are recorded on the data sheet. The difference between the monitor response and the calibration gas value is the CD test result, which is recorded on the data sheet. The $O_2$ monitor calibration must not drift more than an average of 0.5% $O_2$ from the reference standard (calibration gas value) for seven consecutive test days.

### 5.2.2 Calibration Error (CE) Test Protocol

The CE tests will be conducted separately for each monitor while the system is operating under normal conditions during the 7-day CD test period. The CE test will be conducted by determining the monitor response to the injection of zero-level, mid-level, and high-level calibration gases at the sample probe. The calibration gas values used for each of the monitors are presented in Table 5-3. The test protocols are summarized below.

#### 5.2.2.1 Carbon Monoxide Monitor

During the CO CE test, the CEMS test operator obtains the CO gas standard concentrations from the gas cylinders, records the concentration values on the data sheet, and confirms that the concentrations are within the ranges shown in Table 5-3 for conducting the CE test.

The zero-level, mid-level, and high-level calibration gases are injected non-consecutively at each of the three measurement points and the monitor responses to the gases are recorded on the data sheet. (Note that the monitor responses recorded are not corrected to 7% oxygen.) The difference between the monitor response and the calibration gas value at each level is calculated and recorded on the data sheet. After completing three runs each of the low, mid, and high span ranges, the mean difference of the readings at the low-level, mid-level, and high-level gases is calculated and entered on the data sheet. The CE result is presented as a percent of span, which is calculated by dividing the mean difference at each level by the appropriate monitor span (200 ppmv for the low range and 3,000 ppmv for the high range). The mean difference between the CEMS and reference values at all three test points must be no greater than 5 percent of span value.

#### 5.2.2.2 Oxygen Monitor

The CEMS test operator obtains the $O_2$ gas standard concentrations (zero-level, mid-level, and high-level) from the gas cylinders, records the concentrations on the data sheet, and confirms that the concentrations are within the ranges shown in Table 5-3 for conducting the CE test.

The zero-level, mid-level, and high-level calibration gases are injected non-consecutively at each of the three measurement points and the monitor responses to the gases are recorded on the data sheet. The difference between the monitor response and the calibration gas value at each level is calculated and recorded on the data sheet. After completing three runs each of the low, mid, and high span ranges, the mean difference of the readings at the low-level, mid-level, and high-level gases is calculated and entered on the data sheet. The CE result is presented as a percent of span, which is calculated by dividing the mean difference at each level by the monitor span (25% $O_2$). The mean difference between the CEMS and reference values at all three test points must be no greater than 0.5 percent of span.

### 5.2.3 Relative Accuracy (RA) Test

The RA tests will be conducted during the CD test period. This test is conducted using a specified reference method (RM) test to determine an actual or true value. A total of 12 RA tests will be conducted. Hexion can reject a maximum of three tests to demonstrate compliance with the RA criteria as long as nine readings are used to determine the RA. All data points will be reported.

The RA testing will be conducted on a CO reading that has been corrected to 7% $O_2$ such that a separate RA test does not have to be conducted on the $O_2$ monitor.

The RA test will be performed on the CO monitor using EPA Method 10, 10A, or 10B (40 CFR 60, Appendix A) as the RM. Since the CO monitor is a NDIR monitor, an alternative interference trap will be used for the RM test as specified in Section 10.1 of the Performance Specification 2 of 40 CFR 60, Appendix B.

The RM and CO monitor measurement will be consistent on a moisture and % $O_2$ basis. The CO monitor response will be compared against the corresponding RM value and the results will be summarized. The mean of the differences between the RM value and the CO monitor response, the standard deviation, the confidence coefficient, and the CO monitor RA will be calculated using the equations in Section 8.0 of Performance Specification 2 of 40 CFR 60, Appendix B. For the CO monitor, the relative accuracy shall be no greater than 10 percent of the mean value of the RM mean or 5 ppm, whichever is greater.

### 5.2.4 Response Time (RT) Test

The RT tests will be conducted during the CD test period. The CO and the $O_2$ monitors will be tested at zero-level and high-level calibration gas values. The entire CEMS will be checked including the sample extraction, transport, and conditioning apparatus, and the gas analyses and data recording systems.

#### 5.2.4.1 Carbon Monoxide Monitor

A zero-level calibration gas will be introduced into the system as close to the sample probe as practical. Once the system has stabilized (i.e. no change greater than 1% of the full scale for 30 seconds), the monitor will be switched to monitor the combustion gas and the time required for the system to reach 95% of the final stable value will be recorded. This time is called the upscale RT.

Once a stable value has been attained, the high-level calibration gas will be introduced at the same point. Once a stable value has been attained, the monitor will be switched back to the combustion gas and the time required for the system to reach 95% of the final stable value will be recorded. This is called the downscale RT. This procedure will be repeated three times. Once the tests are completed, an average RT will be calculated for both the upscale and downscale RT's. The longer of these two means is the RT for the system. The response time for the CO monitor must not exceed 2 minutes

In some cases, the combustion gas concentration is approximately equal to the zero-level calibration gas concentration such that there may be no noticeable step change in the system reading once the monitor is switched from monitoring the calibration gas to monitoring the combustion gas. In these cases, an upscale reading cannot be obtained. If this occurs, the RT from the downscale measurement will be used to determine compliance with the RT performance criteria.

#### 5.2.4.2 Oxygen Monitor

The RT test will be conducted for the $O_2$ monitor as described above for the CO monitor. The RT test data will be recorded on a data sheet similar to the RT for the CO monitor. The response time for the $O_2$ monitor must not exceed 2 minutes.

### 5.2.5 Pretest Preparation

#### 5.2.5.1 Calibration Gas Cylinder Inventory

Prior to initiating the CEMS PET, a calibration gas inventory will be conducted to verify and document that the required calibration gases are on-site. Table 5-3 lists all calibration gases required for conducting the CEM PET.

#### 5.2.5.2 Installation Verification

Performance Specification 4B suggests that the sampling point be located:

- ≥ 2 equivalent duct diameters downstream from the nearest control device, point of pollution generation or other point at which a change in the pollutant concentration or emission rate may occur.
- ≥ 0.5 equivalent duct diameters upstream from the effluent exhaust or next control device.

NCIN-1 and NCIN-2 sampling points more than exceed the Specification 4B guidance and will provide representative samples of the combustion gas stream from the incinerators. In addition, the sample probe assemblies are located in stacks that are operated under positive pressure and should be free from air in-leakage that could impact combustion gas concentrations.

#### 5.2.5.3 Stratification Test

Stratification tests are not required as part of this evaluation because all points are less than 1.0 meter from the side wall of the duct (paragraph 3.3 of Performance Specification 4B). In addition, NCIN-1 and NCIN-2 combustion gases are thoroughly mixed. The sample point locations are free from air in-leakage. There is no reason to believe that the monitored gases could be stratified to the extent that it would cause failure of the RA test. Hexion will verify this by inspection and record the findings.

#### 5.2.5.4 Reference Method Sampling and Analytical System

A qualified stack sampling contractor using a temporary CEMS will be utilized to conduct the RA test for the CO and $O_2$ monitors. This CEMS will be used to perform the RM identified in Paragraph 4.3 of Performance Specification 4B for CO and Section 3 of Performance Specification 3 for $O_2$. The sample port locations for the RM sampling and analytical system are located in the same areas as the other ports for the installed CEMS.

During the RA test, the combustion gas sample for the RM sampling and analytical system will be collected at 3 points on a measurement line (traverse) over the duct cross section. The 3 traverse points will be located at 16.7%, 50%, and 83.3% of the stack diameters as measured from the inside duct wall. During the test, the sample probe will be located within 1.2 inches of the traverse points and the sample collected for 7 minutes at each point (total of 21 minutes).

### 5.3 AWFCO SYSTEM INTEGRATION AND TESTING

The DCS/VAX includes an AWFCO system that stops the hazardous waste feed when the control system records an operating condition outside the limits necessary to comply with permit conditions. The parameter CMS and CEMS described in this plan are integrated with the AWFCO system. Each AWFCO parameter has an alarm and alarm message associated with it that audibly sounds and logs in the control room when the trip point for that AWFCO parameter is approached. The DCS/VAX compares the

instantaneous or calculated rolling average values, depending on the parameter averaging times, to the corresponding parameter trip set point. Upon exceedence of an interlock set point, the DCS/VAX activates shutoff command to stop the hazardous waste feed. In addition, an AWFCO will trigger if the lower or upper range limits of any CMS instrument is reached or exceeded during a one-minute average. Additional AWFCO trips will occur if any CMS instrument measurement DCS/VAX software is turned off or sends an invalid data input.

As part of the CMS PET, the AWFCO system will be tested. To test the AWFCO system, Hexion will simulate a measurement signal in the DCS/VAX associated with the parameters of the CMS. The simulated CMS measurement signal in the DCS/VAX will be increased or decreased, to the level that trips the AWFCO. The indication that the AWFCO has tripped will be shown as an alarm on the computer screen. Hexion will verify that an AWFCO alarm causes the hazardous waste feed to cease. The proper functioning of the system will be documented. Hexion will also perform invalid data input AWFCO tests. Invalid data inputs occur when the DCS/VAX input measurements are out of range or there is a loss of the instrument measurement signal. Hexion will test the invalid data AWFCO trips by turning off the software measurement loop causing a invalid data state.

### 5.4 DATA QUALITY OBJECTIVES

Hexion expects the data measured by the continuous monitoring devices to be precise, accurate, and complete. The CMS will be calibrated using manufacturer's procedures or standard EPA accepted procedures prior to the conducting the Miniburn/CPT. The expected accuracies of the continuous monitoring instruments will be presented in the CMS PEP prepared in accordance with Section 63.8(d) of the MACT General Provisions.

**Table 5-1. Summary of CMS Instrumentation Calibration Procedures**

| Parameter | Instrument Type | Calibration Procedure |
|---|---|---|
| Liquid Feed Rate (lbs/hr) | Mass flow meter | Fill with process fluid; block in flow meter; electronically zero the instrument. |
| Temperature (°F) | Thermocouples | Check thermocouple type and condition. Use a standard thermocouple simulator to generate a millivolt signal from the ANSI standard thermocouple tables corresponding to a given temperature. . At the thermocouple well, generate a signal according to process value listed on the master calibration sheet back to the transmitter or switch, and verify readings on the DCS. Check @ 0%, 25%, 50%, 75% and 100% of the span. Record all as-found and as-left data on the master calibration sheet. Verify that the DCS readout is the same as test value. If an error is detected greater than the manufacture accuracy full range specification, check condition of all wiring and connections, check the transmitter or switch directly, make any repairs or replacements necessary to bring the reading back into tolerance. Record all repairs and replacements on the master calibration sheet. |
| Stack Flow (acfm) | Differential Pressure | Pull and clean the flow element. At the transmitter, generate a signal to the transmitter differential high side cell according to process value listed on the master calibration sheet back to the transmitter or switch, and verify readings on the DCS. Check @ 0%, 25%, 50%, 75% and 100% of the span. Record all as-found and as-left data on the master calibration sheet. Verify that the DCS readout is the same as test value. If an error is detected greater than the manufacture accuracy full range specification, check condition of all wiring and connections, check the transmitter or switch directly, make any repairs or replacements necessary to bring the reading back into tolerance. Record all repairs and replacements on the master calibration sheet. |

**Table 5-1. Summary of CMS Instrumentation Calibration Procedures**

| Parameter | Instrument Type | Calibration Procedure |
|---|---|---|
| Flow Rate (gpm) | Flow Meters /Transmitters | Inspect flow head element and record condition. To calibrate the transmitter, use a calibrator to simulate the electronic flow signal from the sensor element. Generate a signal according to process value listed on the master calibration sheet and check electronically @ 0%, 25%, 50%, 75% and 100% of the span. Adjust electronically if any drift is found. Record as-found and as-left measurement reading on the master calibration sheet. Verify that the DCS readout is same as test value. If an error is detected greater than the manufacture accuracy full range specification, check condition of all wiring and connections, check the transmitter or switch directly, make any repairs or replacements necessary to bring the reading back into tolerance. Record all repairs and replacements on the master calibration sheet. |
| pH | pH probe and transmitter | Take readings with three buffer solutions of low and high values. Adjust the low and high values to match actual pH values. (Buffer solutions of low and high values are not the same as Zero and Span.) Use pH medium buffer solution to check for linearity test value. Record as-found and as-left measurement reading on the master calibration sheet. Verify that the DCS readout is the same as test value. If an error is detected greater than the manufacturer accuracy full range specification, check condition of all wiring and connections, check the transmitter or switch directly, make any repairs or replacements necessary to bring the reading back into tolerance. Record all repairs and replacements on the master calibration sheet. |

**Table 5-1. Summary of CMS Instrumentation Calibration Procedures**

| Parameter | Instrument Type | Calibration Procedure |
|---|---|---|
| Pressure (in. of w.c.) | Diff. Pressure Transmitters | Use a standard pressure calibrator to generate a signal corresponding to the pressure signal data listed on the master calibration sheet, typically, 5 points. Verify that the DCS readout is the same as test value. Record as-found and as-left calibration measurement readings on the master calibration sheet. If an error is detected greater than the manufacture accuracy full range specification, check condition of all wiring and connections, check the transmitter or switch directly, make any repairs or replacements necessary to bring the reading back into tolerance.<br>Record all repairs and replacements on the master calibration sheet. |

**Table 5-2. CEMS Performance Criteria [a]**

| Monitor/Test | Performance Criteria | Reference | Notes |
|---|---|---|---|
| **Carbon Monoxide Monitor** | - | - | |
| Calibration Drift | ≤ 3 % of span | PS 4B, 4.2 | For 6 out of 7 days; low and high range |
| Calibration Error | ≤ 5 % of span | PS 4B, 4.4 | At all three test points |
| Response Time | ≤ 2 minutes | PS 4B, 4.5 | |
| Relative Accuracy | ≤ 10 % of RM Mean □ | PS 4B, 4.3 (PS 4A, 2.5) | or 5 ppm, whichever is greater [b] |
| **Oxygen Monitor** | - | - | |
| Calibration Drift | ≤ 0.5 % $O_2$ | PS 4B, 4.2 | For 7 consecutive days |
| Calibration Error | ≤ 0.5 % $O_2$ | PS 4B, 4.4 | At all three test points |
| Response Time | ≤ 2 minutes | PS 4B, 4.5 | Longest of the upscale and downscale averages |
| Relative Accuracy | NA | BIF [c] | Incorporated into CO RA test |

PS - Performance Specification, RM - Reference Method

Notes:

[a] Original reference for performance criteria is Performance Specification 4B.

[b] If the average concentration of CO in the emissions is < 10 ppmv (I.e., < 10 % of the 100 ppmv standard), compliance with the RA criteria has been demonstrated if the RM demonstrates that CO emissions are < 10 ppmv.

[c] 40 CFR 266, Appendix IX, Paragraphs 2.1.4.6 and 2.1.5.3

**Table 5-3. CEMS Performance Evaluation Test Calibration Gas Requirements**

| TEST/MONITOR | UNITS | CALIBRATION GAS CONCENTRATIONS [a] | | |
|---|---|---|---|---|
| | | Zero | Mid-Level | High-Level |
| **Calibration Drift and Response Time Tests[b]** | | | | |
| CO - Low span | ppmv | 0-40 | NA | 100 - 180 |
| CO - High span | ppmv | 0-600 | NA | 1,500 - 2,700 |
| $O_2$ | vol% | 0-5 | NA | 12.5 - 22.5 |
| **Calibration Error Test[c]** | | | | |
| CO - Low span | ppmv | 0 -40 | 60 - 80 | 140 - 160 |
| CO - High span | ppmv | 0 -600 | 900 - 1,200 | 2,100 - 2,400 |
| $O_2$ | vol% | 0 -2 | 8 - 10 | 14 - 16 |

Notes:
[a] A copy of the suppliers certificate of analysis must be provided for each gas cylinder. The gas concentrations for the calibration error tests must be certified by the suppliers according to EPA Protocol 1.
[b] Cal gas concentration ranges per 40 CFR 266 Appendix IX, Section 2.0, Article 2.1.4.2.1 and 40 CFR 266 Appendix IX, Section 2.0, Article 2.1.4.2.2
[c] Cal gas concentration ranges per 40 CFR 60, Appendix B, Performance Specification 4B

## 6.0 TEST SCHEDULES

The performance evaluation of the parameter CMS will be completed in conjunction with the testing of the CEMS. The testing of both parameter CMS and the CEMS is expected to take approximately 7 days on-site and preparation will begin approximately 3 weeks prior to the CPT.

# 7.0 QUALITY ASSURANCE

Pursuant to Section 63.8(e)(3) of the MACT General Provisions, the CMS PETP must include an internal and external QA program. The QA programs are described below:

## 7.1 INTERNAL QA

The quality of data generated by the system will be assured by implementing internal quality control procedures. The internal QA program will include the activities planned by routine operators and analysts to provide an assessment of CMS performance. The routine procedures include the following:

- Testing and other quality assurance checks of parameter CMS and CEMS following procedures summarized by this test plan and specified in the CMS PEP. In addition to this CMS PETP, the CMS PEP will be prepared pursuant to Section 63.8(d) of the MACT General Provisions and will be finalized and integrated into Hexion's Operating Record by the HWC MACT compliance date or the submission of a Notification of Compliance (NOC) or a Documentation of Compliance (DOC), whichever is earlier.
- Field verification of the CMS instrument location, condition, and installation.
- Daily audit of the CEMS components and operating parameters. Corrective action will be taken as needed to remedy malfunctions or abnormal operating conditions.
- Daily calibration drift checks on the CEMS. The results of the calibration drift check will be reviewed daily and the monitoring instruments will be adjusted for drift as appropriate.

## 7.2 EXTERNAL QA

Hexion's external QA program shall include systems audits that include the opportunity for on-site evaluation of instrument calibration, data validation, sample logging, and documentation of quality control data and field maintenance activities. The QA program implementation will verify conformance via:

- Reviews of calibration procedures in the CMS PEP
- Reviews of data sheets to ensure completeness and accuracy
- Examinations of facility records documenting the data verification and data reduction/calculation procedures performed for compliance purposes.

The Administrator may perform similar verification of conformance to the QA Program by a similar review process.

In addition, the quality of data generated by the CMS will be assured by implementing the quality control procedures presented in the CMS PEP. This program addresses all aspects of quality control for NCIN-1 and NCIN-2 process parameter monitors and the continuous emission monitors.

## 8.0 DOCUMENTATION

Results of the performance evaluations will be summarized on data sheets. All data sheets, calculations, CMS system data records, and calibration or reference material certification will be included in CMS PET Report. This report will be included as part of the NOC and will be maintained as part of the Operating Record.

# APPENDIX A: EXAMPLE CALIBRATION DATA SHEETS

# EXHIBIT A-1

## EXAMPLE DATA SHEET FOR CONDUCTING THE CARBON MONOXIDE MONITOR SEVEN DAY CALIBRATION DRIFT TEST

Source: _____ Operator: _____

Location: _____ Low Range (ppmv): _____

Monitor: _____ High Range (ppmv): _____

Serial Number: _____

| Calibration Gas Category | Day | Date | Operator Initials | Calibration Gas Concentration (ppm) | (a) Monitor Response (ppm) | CD Result Difference (±ppm) | (b) % of Span | (c, d) Pass/Fail |
|---|---|---|---|---|---|---|---|---|
| Zero Level | 1 | | | | | | | |
| | 2 | | | | | | | |
| | 3 | | | | | | | |
| | 4 | | | | | | | |
| | 5 | | | | | | | |
| | 6 | | | | | | | |
| | 7 | | | | | | | |
| Low Span Level | 1 | | | | | | | |
| | 2 | | | | | | | |
| | 3 | | | | | | | |
| | 4 | | | | | | | |
| | 5 | | | | | | | |
| | 6 | | | | | | | |
| | 7 | | | | | | | |
| High Span | 1 | | | | | | | |
| | 2 | | | | | | | |
| | 3 | | | | | | | |
| | 4 | | | | | | | |
| | 5 | | | | | | | |
| | 6 | | | | | | | |
| | 7 | | | | | | | |

a) Monitor response reading before correction to 7% oxygen.
b) % of Span = Difference/Span x 100
c) The acceptance criteria is ≤ 3% of span (≤ 6 ppmv for the low range and ≤ 90 ppmv for high range)
d) The CO monitor must pass the daily CD test 6 out of the 7 days (Section 4.2 of Performance Specification 4B).

# Exhibit A-2

## EXAMPLE DATA SHEET FOR CONDUCTING THE OXYGEN MONITOR SEVEN DAY CALIBRTION DRIFT TEST

Source: _____ Operator: _____

Location: _____ Range (vol%): _____

Monitor: _____

Serial Number: _____

| Calibration Gas Category | Day | Date | Operator Initials | Calibration Gas Concentration (vol%) | Monitor Response (vol%) | CD Result Difference (±vol%) | (a) Pass/Fail |
|---|---|---|---|---|---|---|---|
| Zero Level | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |
| | 4 | | | | | | |
| | 5 | | | | | | |
| | 6 | | | | | | |
| | 7 | | | | | | |
| Span Level | 1 | | | | | | |
| | 2 | | | | | | |
| | 3 | | | | | | |
| | 4 | | | | | | |
| | 5 | | | | | | |
| | 6 | | | | | | |
| | 7 | | | | | | |

a) The acceptance criteria is ≤ 0.5 vol%.

**EXHIBIT A-3**

# EXAMPLE DATA SHEET FOR CONDUCTING THE CARBON MONOXIDE MONITOR CALIBRATION ERROR TEST

Source: _____ Date: _____

Sample Probe Location: Exhaust Duct Time: _____

System No. _____ Operator: _____

CO Monitor: _____ Serial No._____
(Manufacturer and model number)

Span: 0-200 ppm
0-3000 ppm Tag No._____

| Run Number | Cylinder I.D. No. | Calibration Value (ppm) | Monitor Response (ppm) | Difference (ppm) Zero/Low | Mid | High |
|---|---|---|---|---|---|---|
| **Low Span (0 - 200 ppm)** | | | | | | |
| 1 - Zero | | | | | | |
| 2 - Mid | | | | | | |
| 3 - High | | | | | | |
| 4 - Mid | | | | | | |
| 5 - Zero | | | | | | |
| 6 - High | | | | | | |
| 7 - Zero | | | | | | |
| 8 - Mid | | | | | | |
| 9 - High | | | | | | |
| | | | Mean Difference (ppm) = | | | |
| | | | Low Span Calibration Error (% of span) = | | | |
| **High Span (0 - 3000 ppm)** | | | | | | |
| 1 - Zero | | | | | | |
| 2 - Mid | | | | | | |
| 3 - High | | | | | | |
| 4 - Mid | | | | | | |
| 5 - Zero | | | | | | |
| 6 - High | | | | | | |
| 7 - Zero | | | | | | |
| 8 - Mid | | | | | | |
| 9 - High | | | | | | |
| | | | Mean Difference (ppm) = | | | |
| | | | High Span Calibration Error (% of span) = | | | |

## EXHIBIT A-4

## EXAMPLE DATA SHEET FOR OXYGEN MONITOR CALIBRATION ERROR TEST

Source: _____ Date: _____

Sample Probe Location: Exhaust Duct Time: _____

System No. _____ Operator: _____

$O_2$ Monitor: _____ Serial No._____

(Manufacturer and model number)

Span: 0-25 vol% Tag No._____

| Run Number | Cylinder I.D. No. | Calibration Value (%) | Monitor Response (%) | Difference (%) | | |
|---|---|---|---|---|---|---|
| | | | | Zero | Mid | High |
| 1 - Zero | | | | | | |
| 2 - Mid | | | | | | |
| 3 - High | | | | | | |
| 4 - Mid | | | | | | |
| 5 - Zero | | | | | | |
| 6 - High | | | | | | |
| 7 - Zero | | | | | | |
| 8 - Mid | | | | | | |
| 9 - High | | | | | | |
| | | | Mean Difference (%) = | | | |

# Exhibit A-5. Example Data Sheet
## Response Time

Date: _____

Inspector: _____

CEMS Train: A B (circle one) Analyzer: CO or $O_2$ (circle one)

Analyzer Manuf. and Model #: _____

Analyzer Serial #: _____

Record

**ZG** = Zero Calibration Gas = _____ ppmv or % (circle one) Cylinder # = _____

**SG** = Span Calibration Gas = _____ ppmv or % (circle one) Cylinder # = _____

Calculate

**Response Time (RT) = Time required to reach 95% of stable value**

**Approx. $RT_U$ Stable Value = .95 X SG =** _____ ppmv or % (circle one)

**Approx. $RT_D$ Stable Value = .95 X ZG =** _____ ppmv or % (circle one)

| | Action | | 95% of Stable Value (ppm or %) | Response Time for 95% of Final Value: Upscale (seconds) | Response Time for 95% of Final Value: Downscale (seconds) |
|---|---|---|---|---|---|
| 1 | Apply **Zero Gas** until monitor reads stable for 30 seconds. Stable is considered +/- 1% of span. | | | | |
| 2 | Apply a step change by introducing the **Span Gas**. Record the time to reach **$RT_U$**. | **$RT_U$** | | | |
| 3 | Reintroduce the **Zero Gas** and wait for a stable reading before recording the **$RT_D$** | **$RT_D$** | | | |
| 4 | Apply a step change by applying **Span Gas**. Record the time to reach **$RT_U$**. | **$RT_U$** | | | |
| 5 | Apply a step change by applying **Zero Gas**. Record the time to reach **$RT_D$**. | **$RT_D$** | | | |
| 6 | Apply a step change by applying **Span Gas.** Record the time to reach **$RT_U$**. | **$RT_U$** | | | |
| 7 | Apply a step change by applying **Zero Gas**. Record the time to reach **$RT_D$**. | **$RT_D$** | | | |
| | **Average Response Time (120 sec max) =** | | | | |

The slower of the two means ($RT_D$ and $RT_U$) is the response time

**Exhibit A-6**

## EXAMPLE DATA SHEET FOR CONDUCTING THE QUARTERLY ABSOLUTE CALIBRATION AUDIT (ACA) OR RELATIVE ACCURACY TEST AUDIT (RATA) FOR CARBON MONOXIDE/OXYGEN MONITORS

Source: _____ Date: _____

Sample Probe Location: Exhaust Duct Operator: _____

CO Monitor: _____ Serial No. _____

Span: 0-200 ppm, 0-3000 ppm Tag No._____

O2 Monitor: _____ Serial No. _____

Span: 0-25 % Tag No._____

| | Carbon Monoxide (Low Range) | | | Carbon Monoxide (High Range) | | | Oxygen | | |
|---|---|---|---|---|---|---|---|---|---|
| Measurement | CEMS Response (ppm) | Calibration Gas Concentration (ppm) | Difference (ppm) | CEMS Response (ppm) | Calibration Gas Concentration (ppm) | Difference (ppm) | CEMS Response (%) | Calibration Gas Concentration (ppm) | Difference (%) |
| 1 | | | | | | | | | |
| 1 | | | | | | | | | |
| 1 | | | | | | | | | |
| 2 | | | | | | | | | |
| 2 | | | | | | | | | |
| 2 | | | | | | | | | |
| Arithmetic Mean (ppm) [a] | | | | | | | | | |
| Relative accuracy (% of RM) [a] | | | | | | | | | |

# Exhibit A-7
# CPMS Instrument Calibration Data Sheet

| Instrument Information | Specification |
|---|---|
| Instrument ID: | Range/Set point |
| Date Calibration Due | Instrument Tolerance |
| Instrument Name | Process Tolerance |
| Work Order Number | |

| Remarks/Comments |
|---|
| |
| |
| |
| |

**Calibration Data**

| Parameters | Units | STD. Reading | Instrument Tolerance | As Found Reading | Deviation | Final Reading |
|---|---|---|---|---|---|---|
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |

**Reference Instrument Used**

| ID Number | Description | Manufacturer | Calibrated Date | Calibrated Due Date |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |

| Action Taken | Certification & Completion Report | | |
|---|---|---|---|
| Comply with specs | Done By: | Date: | Hours |
| Calibrated | | | |
| Out of Service | Supervisor: | Date: | Entry Check: |
| Not Found | | | |

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AI# 87883

Hexion Specialty Chemical
16122 River Rd
Lot 3
Norco, La 70079
St. Charles Parish

Material associated with Trial Burn/Mact for Public Review; {Scheduled for 03/30/2009 to 4/13/2009}

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